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PRACTICAL CHEMISTRY.

PRACTICAL CHEMISTRY.

THE OWENS COLLEGE JUNIOR COURSE

OF

PRACTICAL CHEMISTRY.

BY

FRANCIS JONES, F.R.S.E., F.C.S.,

Chemical Master in the Grammar School, Manchester.

WITH A PREFACE BY

SIR H. E. ROSCOE, F.R.S.

London:

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1887.

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PREFACE.

THIS little book contains a short description of a course of practical chemistry, which an experience of many years has proved suitable for those commencing the study of the science. The work is, however, not intended to supplant, but rather to supplement instruction given by the Teacher, as it points out the principles of the various processes and names the various reactions without entering into the details of the necessary manipulation, which after all can only be satisfactorily learnt by practical demonstration. The subject-matter has been carefully compiled, under my supervision, by Mr. Francis Jones, F.R.S.E., formerly Junior Demonstrator in this Laboratory, and many improvements in the analytical tables are due to Mr. Schorlemmer, F.R.S.. the Senior Demonstrator.

To those about to use the book, I would give the following advice :–

39490

PRACTICAL CHEMISTRY.

PART IV.

PAGE
Grouping and Reactions of the Inorganic Acids 97
Separation of Inorganic Acids 115
Grouping and Reactions of the Organic Acids 118
Tests for Organic Alkaloids 125
Separation of Organic Acids 134

PART V.

Grouping and Reactions of the Rare Metals 136

PART VI.

Examples of Quantitative Analysis 150

APPENDIX.

A. Table of the Elements 173
B. Table of Weights and Measures 176
C. Treatment of Silver Residues 177
D. Treatment of Platinum Residues 180
Questions and Exercises 181

INDEX 199

JUNIOR COURSE OF LABORATORY PRACTICE.

PART I.

PREPARATION OF APPARATUS, GASES, &c.

1. *Glass stirring-rods.*

Divide a piece of glass rod into several pieces about two decimetres in length. This is done by filing the glass rod at each place where it is to be cut off, with a three-cornered file, and then snapping it across. Knock off any projecting pieces of glass which may be left at the newly-cut edges, and then hold each end of the rod in the flame of a Bunsen lamp until the sharp edges are fused and rounded. The glass rods thus made serve for stirring liquids, &c.

2. *A wash bottle.*

Soften a cork* by gently rolling it under the foot, and fit it air-tight into the neck of a flask about one litre capacity. Then, by means of a round file, bore two holes in the cork about three millimetres in diameter, and running parallel to each other and to the longer axis of the cork. Next obtain two pieces of glass tubing of the same diameter, one three decimetres long and the other half that length. Hold one end of the longer tube in the Bunsen flame until the opening contracts considerably (but take care not to seal it up entirely), and then bend it about half a decimeter from the end, as shown in the figure. This is done by holding the glass tube horizontally in a common batswing gas jet flame, turning it round so as to heat all parts equally, and bending to the proper angle as soon as it feels sufficiently soft. Now round the edge of the wide end of the tube by holding it in the flame till it softens, and when cold fit it into the cork. In a similar way round both ends of the shorter piece of tubing, bend to the angle shown in the figure, and fit into the other hole. Clean out the flask and tubes thoroughly, rinse with distilled water, and then fill up with distilled water.

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* A doubly-bored india-rubber stopper may be used instead of a cork

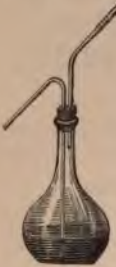

FIG. 1.

3. *Preparation of oxygen from mercuric oxide.*

When mercuric oxide is heated it is decomposed into mercury and oxygen.

Mercuric oxide yields mercury and oxygen.

$$^{*}\ HgO = Hg + O$$

$$216 = 200 + 16$$

---

* For the explanation of these symbols a larger work must be consulted. See p. 13 of Roscoe's "Elementary Chemistry."

Place a small quantity of mercuric oxide in a dry test tube, and heat it over a Bunsen lamp. The substance darkens in colour, and a ring of minute globules of mercury soon forms on the cool part of the tube. That the tube contains oxygen may be shown by plunging a glowing chip of wood into it, and observing that the wood will be rekindled. It is advisable to keep the thumb loosely on the mouth of the test tube to prevent the escape of oxygen by diffusion.

4. *Preparation of oxygen from potassium chlorate.*

Potassium chlorate when heated is decomposed into oxygen and potassium chloride.

Potassium chlorate yields potassium chloride and oxygen:

$$\begin{aligned} KClO_3 &= KCl + O_3. \\ 122{\cdot}6 &= 74{\cdot}6 + 48. \end{aligned}$$

Place a few crystals of potassium chlorate in a dry test tube, and heat gently. The salt soon fuses and then begins to effervesce, giving off oxygen, which may be recognized, as before, by its power of rekindling a glowing chip. When oxygen ceases to be evolved, the residue is a white salt called potassium chloride.

5. *Tests for potassium chlorate and chloride.*

A solution of potassium chlorate is *not precipitated* by a solution of silver nitrate; but potassium chloride *is precipitated*, silver chloride being formed.

Potassium chloride and silver nitrate yield potassium nitrate and silver chloride:

$$\begin{aligned} KCl + AgNO_3 &= KNO_3 + AgCl. \\ 74{\cdot}6 + 170 &= 101{\cdot}1 + 143{\cdot}5. \end{aligned}$$

Dissolve a crystal of potassium chlorate in distilled water, then add solution of silver nitrate, no precipitate will be formed. Dissolve also the residue of potassium chloride (obtained in **4**) in distilled water, and add silver nitrate solution. A curdy white precipitate of silver chloride will at once be formed.

6. *Preparation of oxygen from potassium chlorate and manganese dioxide.*

Potassium chlorate when mixed with about one-fifth of its weight of manganese dioxide, gives off its oxygen at a much lower temperature than when heated alone, although the oxide itself remains quite unaltered.

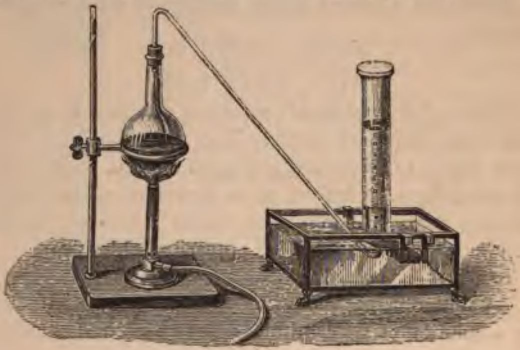

FIG. 2.

Place in a small flask (about 100 cubic centimetres capacity) a mixture of potassium chlorate and manganese dioxide in the proportions already mentioned. Then fit into the neck of the flask a cork through which a bent conducting tube passes, the lower end of which is placed under water in the pneumatic trough. Fill some gas jars with water and invert them in the trough. Support the flask on a retort stand, and heat the mixture (Fig. 2); as soon as heat is applied, bubbles of gas will begin to rise through the water, consisting of air expelled from the flask by the heat. These are allowed to escape, and the oxygen, which soon begins to come off abundantly, is collected by placing the inverted bottles over the end of the conducting tube, and thus allowing the bubbles of oxygen to ascend into the bottles and displace the water. As soon as the first bottle is filled with oxygen, place a shallow earthenware tray under the neck, and remove it from the trough, taking care that the tray contains enough water to prevent the escape of the gas. When four bottles have been filled in this manner, remove the flask and conducting tube and allow the former to cool.

7. *The residue from the preparation of oxygen from potassium chlorate and manganese dioxide is potassium chloride, and unaltered manganese dioxide.*

Dissolve in water the residue in the flask from the preparation of oxygen, filter from the insoluble manganese dioxide, and evaporate the filtrate in a porcelain basin to a small bulk; on cooling, crystals of potassium chloride will separate out. Pour off the mother liquor, dry the crystals between filter paper, and keep them in a small bottle for future experiments.

8. *Combustions in oxygen.*

When a taper is burned in oxygen, the carbon which it contains unites with the oxygen, forming carbon dioxide (carbonic acid gas), and the hydrogen also unites with the oxygen, forming hydrogen monoxide (water).

Carbon and oxygen yield carbon dioxide:

$$C + O_2 = CO_2.$$
$$12 + 32 = 44.$$

Hydrogen and oxygen yield water:

$$H_2 + O = H_2O.$$
$$2 + 16 = 18.$$

Place a small piece of taper on an iron wire support, light it, and plunge it into one of the jars of oxygen (prepared in **6**). Observe that it burns much more brightly than in air. Withdraw it, blow the light out, and if only a spark remain on the wick, observe that when again plunged into the oxygen it is at once rekindled. This serves as a convenient test for the presence of oxygen. When solution of calcium hydrate (lime water) is placed in contact with carbon dioxide, calcium carbonate (chalk) and water are formed, and the former, being insoluble in water, is precipitated as a white powder.

Calcium hydrate and carbon dioxide yield calcium carbonate and water:

$$Ca(HO)_2 + CO_2 = CaCO_3 + H_2O.$$
$$74 + 44 = 100 + 18.$$

Prove that the jar in which you have burned the taper contains carbon dioxide, by adding a little clear lime water to it and shaking so as to bring the gas and liquid in contact. A white precipitate will be formed, consisting of calcium carbonate.

9. *When charcoal is burned in oxygen carbon dioxide is produced.* (Compare **8.**)

Place a few pieces of charcoal, about the size of peas, in the deflagrating spoon, hold this in the lamp-flame till the charcoal is just kindled, and then plunge the spoon into a jar of oxygen. The charcoal will burn with great brilliancy, and, if enough oxygen be present, nothing will remain on the spoon but a little white ash (the inorganic matter in the charcoal).

Prove the presence of carbon dioxide (as in **8**) by adding lime water.

A similar union of carbon and oxygen takes place in animals when breathing, but the combustion is slow, not rapid. A portion of the carbon of their bodies unites with the oxygen which they inhale from the air, and carbon dioxide is produced. Observe this by blowing the air from the lungs through a glass tube into some clear lime water, a white precipitate of calcium carbonate soon forms, caused by the union of the carbon dioxide of the breath with the lime. (See Roscoe, p. 11.)

10. *When sulphur is burned in oxygen, sulphur dioxide (sulphurous anhydride) is produced.*

Sulphur and oxygen yield sulphur dioxide :

$$S + O_2 = SO_2.$$
$$32 + 32 = 64.$$

Place a few pieces of sulphur in the deflagrating spoon, heat until the sulphur is melted and takes fire, and then plunge it into a jar of oxygen. Observe that the sulphur, which only burned feebly in air, burns with considerable brightness in oxygen. When it has ceased to burn, remove the spoon, and observe the suffocating odour of the gas which has been produced.

Free acids redden blue vegetable colouring matters.

Show that sulphur dioxide when dissolved in water forms an acid, and on pouring a solution of blue litmus into the wet bottle in which the sulphur was burned, observe that the blue colour is changed to red.

11. *When phosphorus is burned in oxygen, phosphorus pentoxide (or phosphoric anhydride) is produced.*

This is not a gaseous body like the dioxides of sulphur and carbon, but is a white solid substance, which very readily unites with water, forming tri-hydrogen phosphate (tribasic phosphoric acid).

Phosphorus and oxygen yield phosphorus pentoxide :

$$P_2 + O_5 = P_2O_5.$$
$$62 + 80 = 142.$$

When the oxygen has been collected over water, there is always enough moisture in the gas-jar to unite with the pentoxide, and form a solution of phosphoric acid ($H_3 P O_4$), thus :

Phosphorus pentoxide and water yield tri-hydrogen phosphate :

$$P_2O_5 + 3 H_2O = 2 H_3PO_4.$$
$$142 + 54 = 196.$$

Place a small piece of phosphorus (having carefully dried it with filter-paper) on the deflagrating spoon, light it by touching it with a hot wire, and place it in a jar of oxygen. Observe the intensely brilliant light with which it burns, and the dense white fumes of phosphorus pentoxide which are produced. Observe too that in a short time these fumes disappear for the reason already mentioned; and show that an acid is present by pouring in some litmus solution and observing the change of colour from blue to red.

12. *Presence of oxygen in air.*

Oxygen is present in the air diluted with another gas called nitrogen. In one hundred volumes of air, about twenty-one consist of oxygen, and seventy-nine of nitrogen. When substances burn in air they unite with the oxygen in it and produce oxides, just as they do when burned in the pure gas.

Burn a taper, sulphur, and phosphorus in three jars of air, and prove that carbon dioxide, sulphur dioxide, and phosphorus pentoxide are respectively produced. Ascertain this by applying the same tests as in **8. 10.** and **11.**

13. *Preparation of hydrogen.*

By the action of acids on certain metals, such as zinc, magnesium, and iron, hydrogen gas is evolved, a salt of the metal with the particular acid employed, being formed at the same time, thus:

Zinc and sulphuric acid yield hydrogen and zinc sulphate:

$$\begin{array}{l} Zn + H_2SO_4 = H_2 + ZnSO_4 \\ 65{\cdot}2 + \quad 98 \quad = 2 + 161{\cdot}2; \text{ and} \end{array}$$

iron and hydrochloric acid yield hydrogen and ferrous chloride

$$\begin{array}{l} Fe + 2\,HCl = H_2 + FeCl_2 \\ 56 + \quad 73 \quad = 2 + 127. \end{array}$$

Place a few pieces of zinc in a wide-mouthed flask provided with a cork, through which pass two tubes, one a bent conducting tube for leading the gas into the pneumatic trough, the other a funnel tube for supplying acid to the zinc. Fill four jars with water, and collect the gas (which is given off without the aid of heat) in the same way as in the case of oxygen (Fig. 3). Having placed the end of the conducting tube in the pneumatic trough, pour down the funnel tube enough water to cover the zinc completely, and then add sulphuric acid by degrees and cautiously, shaking the flask so as to mix the acid and water. The gas will soon come off with increasing rapidity, but the first portion *must be rejected, as air and hydrogen form an explosive mixture.* It is therefore necessary to wait until all the air has been expelled from the flask by the hydrogen. Before filling the jars, collect a test-tube full of the gas over the pneumatic trough, and apply a light to it (holding the mouth downwards); if

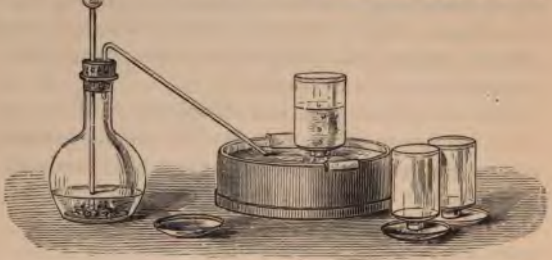

FIG. 3.

it burn quietly, you may safely proceed to fill the gas jars, but if a slight explosion occur, you must wait until another test-tube is collected and found to burn quietly. As soon as this is the case, fill four jars with the gas, and remove them from the trough in the same way as described in **6.**

14. *Production of water by the burning of hydrogen in air.*

Whenever hydrogen burns in air or in oxygen, water is produced.

Hydrogen and oxygen yield water:

$$H_2 + O = H_2O$$
$$2 + 16 = 18.$$

Remove the conducting tube from the hydrogen flask above described (but without removing the cork), and substitute for it a straight piece of glass tube of the same diameter, drawn out at the upper end so as to form a jet (Fig. 4). As all the air has long ago been expelled, the hydrogen issuing from the jet may be safely lighted. Hold over this flame a perfectly dry beaker, or test-glass, and observe the instant deposit of moisture on the sides of the glass. This is caused by the union of the hydrogen with the oxygen of the air contained inside the beaker.

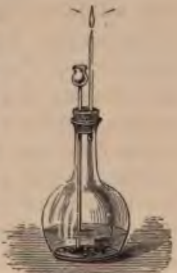

FIG. 4.

15. *The residue from the preparation of hydrogen from zinc and sulphuric acid is zinc sulphate.*

Filter a portion of the contents of the flask used for preparing hydrogen from the undissolved zinc, and evaporate the filtrate to a small bulk. On cooling, crystals of zinc sulphate will be formed, which are drained from the mother liquor, dried between filter paper, and kept in a corked tube or bottle for future examination.

16. *Hydrogen is an inflammable gas.*

Apply a light to one of the jars of hydrogen, and observe that it burns. Notice also the deposit of moisture on the sides of the gas jar caused by the union of the hydrogen with the atmospheric oxygen.

17. *Hydrogen is lighter than air.*

Show this by pouring the contents of one jar, *upwards*, into a jar of air held above the mouth of the hydrogen jar. The hydrogen in virtue of its lightness will leave the lower jar and displace the air from the upper jar. Apply a light to each jar, and observe that the one originally filled with hydrogen no longer contains any, while the other originally filled with air contains hydrogen.

18. *Hydrogen does not support combustion.*

Take another jar of hydrogen, held mouth downwards, and push up into it a lighted taper, supported on a straight wire. The hydrogen will burn at the mouth of the jar, but the taper will be extinguished. The taper may be withdrawn, relighted, and re-extinguished two or three times.

19. *Preparation of nitrogen.*

When phosphorus is burned in air, it unites with the oxygen, forming phosphorus pentoxide (P_2O_5), which dissolves in water, whilst the residue is nitrogen.

Place a small piece of dry phosphorus on a porcelain crucible lid, and fix this on a flat piece of cork, so that the lid may be floated on the water in the pneumatic trough, or other tray containing water. Light the phosphorus, and quickly surround it with a bell-jar, the tubulure of which is closed with a cork (Fig. 5). A portion of the air will first be expelled by the heat, and the phosphorus will continue to burn as long as any oxygen remains in the air of the bell-jar; when that is exhausted it will cease to burn. Observe that the water rises inside the bell-jar, replacing the oxygen, which is no longer gaseous, but has combined with the phosphorus to form phosphorus pentoxide. Wait a few minutes to allow the pentoxide to be completely dissolved by the water. The bell-jar then contains nitrogen.

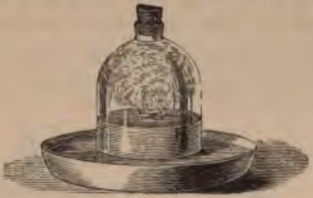

FIG. 5.

20 *Nitrogen does not support combustion, and is not combustible.*

Place a lighted taper in the bell-jar, and observe that the flame is at once extinguished, and that the gas does not burn.

21. *Preparation of nitric acid.*

All nitrates when heated with sulphuric acid are decomposed, nitric acid and a sulphate being formed. Thus, when sulphuric acid acts on potassium nitrate (nitre or saltpetre), nitric acid, and hydrogen potassium sulphate (bisulphate of potash) are produced:

$$KNO_3 + H_2SO_4 = HNO_3 + HKSO_4$$
$$101{\cdot}1 + 98 = 63 + 136{\cdot}1.$$

Put about twenty grams of potassium nitrate (nitre) in a small stoppered retort, place in the tubulure a small funnel, and through this pour the same weight of strong sulphuric acid. Withdraw the funnel without soiling the neck of the retort, place the stopper in the tubulure, and support the retort upon a piece of wire

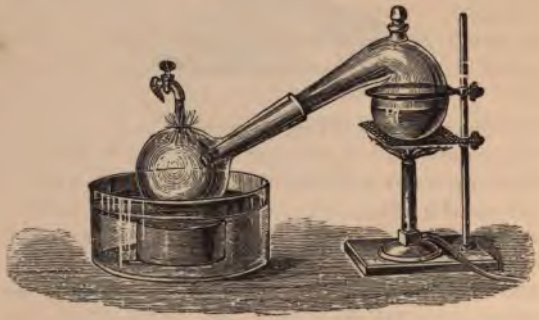

FIG. 6.

gauze placed across the ring of the retort stand. Let the neck of the retort pass into a clean flask, so arranged that a stream of water may fall on it, and thus aid the condensation of the acid (Fig. 6). Now heat the retort, and observe the formation of red fumes (lower oxides of nitrogen) and soon after, the condensation of the nitric acid on the neck of the retort. When the contents of the retort cease to boil, withdraw the lamp, separate the receiving flask containing the nitric acid from the retort, and pour out the contents of the latter into a *dry* porcelain dish. When cold, break up the cake of hydrogen potassium sulphate thus formed, and preserve it for future examination.

22. *Tests for nitric acid.*

(*a*) Add a few drops of the nitric acid prepared as above to a solution of indigo contained in a test-tube. The blue colour will speedily disappear owing to the oxidizing action of the nitric acid.

(*b*) Place a few bits of copper turnings in a test-tube, cover them with water, and add a little nitric acid. The copper soon begins to dissolve, forming a blue solution, and at the same time brown vapours fill the test-tube. In this case also the nitric acid acts as an oxidizing agent, forming copper nitrate, and red fumes of the oxides of nitrogen are given off. For explanation see **30** and **31.**

(*c*) Add a few drops of nitric acid to a little water contained in a test-tube, and then add some strong sulphuric acid, and shake until the liquids are thoroughly mixed. Allow this mixture to cool *completely*, and then pour gently on to the surface of the liquid a solution of ferrous sulphate prepared by dissolving a few crystals of the salt in water. This solution is lighter than the other, and if poured on gently will form a layer of liquid resting on the heavier sulphuric acid, and a black ring will form where the two liquids meet. This is caused by the liberation of nitrogen dioxide (by the action of the $FeSO_4$ on the HNO_3), which forms a dark-coloured compound with the ferrous sulphate. On shaking the tube, nitrogen dioxide will escape with effervescence, and the black ring will disappear.

Retain a portion of nitric acid for **27.**

23. *Tests for nitrates.*

The same reactions may be used to test for combined nitric acid (*i.e.* nitrates), but sulphuric acid must first be added to liberate it.

Repeat the tests *a*, *b*, and *c* (**22**), substituting for the nitric acid a solution of potassium nitrate in water, to which a few drops of strong sulphuric acid have been added.

24. *Preparation of ammonia.*

When caustic alkalies act on salts of ammonium, ammonia gas is liberated. Thus:

Ammonium chloride and caustic lime yield ammonia, calcium chloride, and water:

$$\begin{array}{ccccccccc} 2\,(NH_4)\,Cl & + & CaO & = & 2\,NH_3 & + & CaCl_2 & + & H_2O \\ 107 & + & 56 & = & 34 & + & 111 & + & 18. \end{array}$$

Place about ten grams of powdered ammonium chloride (sal-ammoniac) and the same weight of powdered lime in a test tube provided with a tightly-fitting cork, through which passes a tube bent as shown in the figure. Place over this an inverted dry gas-jar, and heat the mixture in the test tube. After the air has been expelled, the ammonia gas will come over, and in virtue of its lightness be retained in the jar. When the jar is filled, remove it from the upright tube, place the palm of the hand on the mouth, and immerse the jar (mouth downwards) in the pneumatic trough filled with water: the gas will be rapidly absorbed, and the water will rise so as to fill the jar.

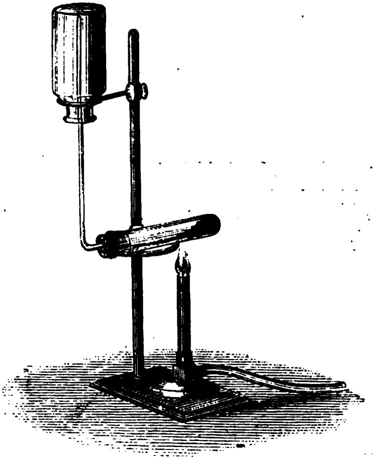

FIG. 7.

Now reverse the conducting tube, and allow the remaining gas to pass into some distilled water contained in a beaker. The gas bubbles will be completely absorbed, and when the gas ceases to be evolved, be careful to withdraw the conducting tube from the liquid, so as to prevent it being sucked back into the hot tube.

25. *Tests for ammonia.*

(*a*) Observe the pungent and very characteristic smell which the solution of ammonia in water possesses.

(*b*) Place a piece of reddened litmus paper *above* the solution, and observe that the vapour given off is able to change the colour from red to blue. Place the litmus paper *in* the solution, and the change will be still more marked.

(*c*) Hold a glass rod which has been dipped in fuming hydrochloric acid over the solution of ammonia : it will at once form white fumes of ammonium chloride, caused by the union of the acid and *volatile alkali*, as ammonia is sometimes called.

Hydrochloric acid and ammonia yield ammonium chloride :

$$\begin{array}{ccccc} HCl & + & NH_3 & = & NH_4Cl. \\ 36{\cdot}5 & + & 17 & = & 53{\cdot}5. \end{array}$$

26. *The residue from the preparation of ammonia from ammonium chloride and lime is calcium chloride.*

Dissolve a portion of the residue contained in the flask used to prepare ammonia, in water, filter from excess of lime, and evaporate the solution to dryness. A white salt results, which is detached from the evaporating basin, and preserved in a well-stoppered bottle for future examination.

27. *Preparation of ammonium nitrate.*

Nitric acid, when neutralized with ammonia, yields ammonium nitrate and water:

$$HNO_3 + (NH_4)HO = (NH_4)NO_3 + H_2O.$$
$$63 \quad + \quad 35 \quad = \quad 80 \quad + \quad 18.$$

Place some nitric acid (**21**) in an evaporating basin, and dilute with twice its bulk of water; add ammonia solution (**24**) cautiously, and with constant stirring until a drop of the liquid ceases to colour litmus paper red. Observe that as the ammonia is added the red colour caused by the nitric acid disappears, and a point is reached when the liquid neither reddens litmus paper nor turns it blue. When this is the case the acid is said to be exactly neutralized, and the addition of ammonia must be stopped. Evaporate the solution of ammonium nitrate thus obtained, until all the water is expelled; when this is the case, withdraw the lamp and allow to cool; a cake of fused ammonium nitrate is then obtained. If the salt does not solidify on cooling, heat must be again applied.

28. *Preparation of nitrogen monoxide (nitrous oxide).*

When ammonium nitrate is heated, nitrogen monoxide and water are produced. Thus:

$$(NH_4)NO_3 = N_2O + 2\,H_2O.$$
$$80 \quad = \quad 44 \quad + \quad 36.$$

Break the cake of ammonium nitrate (**27**) into small pieces, and introduce them into a small dry flask provided with a conducting tube as in **6.** Heat gently, and after allowing the air in the flask to be expelled, begin to collect the gas which is produced. As this gas is soluble to a considerable extent in cold water, it is better to fill the pneumatic trough with warm water, in which the gas is much less soluble. Collect four jars of the gas, and then withdraw the conducting tube from the trough. Do not heat till all the ammonium nitrate is decomposed, as towards the close of the evolution of gas the decomposition sometimes becomes complex, and other gases are generated so rapidly that an explosion may occur.

29. *Properties of nitrous oxide.*

Combustible substances burn in nitrogen monoxide almost as brightly as in oxygen. They decompose the gas, uniting with its oxygen to form oxides, and leaving unaltered nitrogen.

Burn a taper, phosphorus, and sulphur in this gas, in the same manner as they were burned in oxygen. The products of combustion are the same, namely, carbon dioxide, phosphorus pentoxide, and sulphur dioxide; but there is in addition a residue of nitrogen.

30. *Preparation of nitrogen dioxide (nitric oxide).*

When nitric acid acts upon metals such as copper or mercury, nitrogen dioxide, water, and a nitrate of the metal used are produced. Thus:

Copper and nitric acid yield nitrogen dioxide, copper nitrate, and water:

$$3\,\mathrm{Cu} + 8\,\mathrm{HNO_3} = 2\,\mathrm{NO} + 3\,(\mathrm{Cu}\,(\mathrm{NO_3})_2) + 4\,\mathrm{H_2O}.$$
$$190{\cdot}5 + 504 = 60 + 562{\cdot}5 + 72.$$

Place some copper turnings in a flask provided with a funnel and conducting tube, as in the figure. Cover the copper with a layer of water, and add nitric acid by degrees until the gas comes off steadily. Fill two jars completely, and a third, half full of the gas, and leave the last on the support in the pneumatic trough.

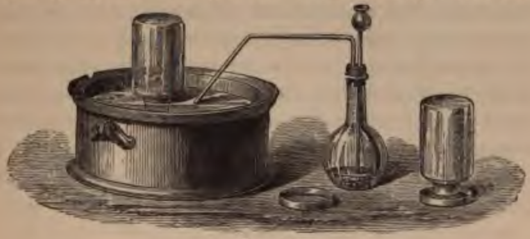

FIG. 8.

31. *Properties of nitrogen dioxide (nitric oxide).*

(*a*) Nitrogen dioxide readily unites with free oxygen, forming higher oxides of nitrogen which have a brown colour, $2\ NO + O = N_2O_3$ and $NO + O = NO_2$. Remove a jar of the gas from the pneumatic trough and expose it to the air. Observe at once the formation of red fumes, consisting chiefly of nitrogen tetroxide, (NO_2). Now dip the mouth of the jar under water, and observe that these fumes disappear owing to their solubility in water, and at the same time observe the rise of water in the jar.

(*b*) Allow a few bubbles of oxygen to pass into the bottle half filled with nitrogen dioxide, and observe as before the formation of red fumes, and the rise of water in the jar as these dissolve.

(*c*) Nitrogen dioxide does not support the combustion of a taper, but phosphorus, when burning very brightly, is not extinguished when placed in the gas. Place a small piece of dry phosphorus in a spoon, light it, and place it in a jar of nitric oxide: it will be extinguished. Now heat it strongly in the gas flame, and again place it, whilst burning brightly, in the gas, and observe that it continues to burn.

32. *Preparation of carbon dioxide* (*carbonic acid gas*).

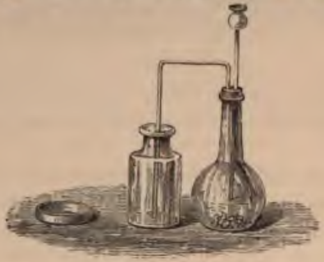

FIG. 9.

When hydrochloric acid acts on calcium carbonate, carbon dioxide, calcium chloride, and water are produced. Thus:

$$CaCO_3 + 2\ HCl = CO_2 + CaCl_2 + H_2O.$$
$$100 \ + \ 73 \ = 44 + 111 + 18.$$

Place some pieces of marble (calcium carbonate) in a flask with funnel and conducting tube, pour some water over it, and then a little hydrochloric acid;

a rapid effervescence will begin, and the gas will be given off copiously. It may be collected over water, but as it is much heavier than air it is best collected by downward displacement. This is done by placing the conducting tube at the bottom of the gas-jars, and allowing the heavy gas to collect in them and displace the air (Fig. 9). As the gas extinguishes flame, the jars are ascertained to be full when a lighted taper placed in the mouth is at once extinguished. When this is the case, cover the jar and substitute another; in this way collect four jars of the gas.

33. *Properties of carbon dioxide.*

(*a*) Carbon dioxide does not support combustion.

Place a lighted taper in the gas: observe that it is at once extinguished.

(*b*) Carbon dioxide precipitates lime water.

Add some clear lime water to a jar of the gas: it is at once rendered milky owing to the formation of calcium carbonate (see **8**). Cover the jar closely with the palm of the hand and shake the bottle: the hand will adhere to the bottle owing to the partial vacuum caused by the absorption of the CO_2 by the lime-water.

(*c*) Carbon dioxide is heavier than air.

Pour the carbon dioxide from one of the gas-jars into a jar of air. The gas is so heavy that it will displace the air from the jar, and that this is the case may be shown by placing a lighted taper in each jar: the one originally full of air will now be found filled with carbon dioxide, whilst the other will, if held mouth downwards for a few minutes, be found to contain only air;

in the former the taper will be extinguished, in the latter it will continue to burn.

34. *Preparation of carbon monoxide (carbonic oxide gas).*

This gas is prepared pure by the action of sulphuric acid upon formic acid, which is decomposed into carbon monoxide and water. Thus:

$$CH_2O_2 = CO + H_2O.$$
$$46 = 28 + 18.$$

It is, however, frequently prepared by the action of sulphuric acid on oxalic acid; but in this case a mixture of equal volumes of carbon dioxide and carbon monoxide is obtained.

Oxalic acid yields carbon dioxide, carbon monoxide, and water:

$$C_2H_2O_4 = CO_2 + CO + H_2O.$$
$$90 = 44 + 28 + 18.$$

Place some crystallized oxalic acid in a small flask provided with a conducting tube (see Fig. 2), cover it with strong sulphuric acid (oil of vitriol), heat gently on a piece of wire gauze, and after allowing the air to escape, collect the gas in the ordinary way at the pneumatic trough. Fill one gas-jar, and a bottle with a neck narrow enough to allow it to be closed with the thumb. Be careful to remove the conducting tube from the pneumatic trough as soon as the bottles are filled with the gas; and allow the contents of the flask to become quite cold before pouring the liquid away.

35. *Properties of carbon monoxide.*

(*a*) The gas burns with a blue flame even when mixed with carbon dioxide, as it is when prepared as above. Apply a light to the wide-mouthed gas-jar containing the mixed gases, and observe the pale blue flame with which the monoxide burns.

(*b*) Pour a small quantity of caustic soda solution into the narrow-necked bottle containing the mixed gases, close it tightly with the thumb, and shake up vigorously without removing the thumb. This will dissolve out the carbon dioxide, which is soluble in caustic soda, hydrogen sodium carbonate being formed, and leave the carbonic oxide. Thus:

$$\begin{array}{ccccccccc} CO_2 & + & CO & + & NaHO & = & NaHCO_3 & + & CO. \\ 44 & + & 28 & + & 40 & = & 84 & + & 28. \end{array}$$

Invert the bottle, mouth downwards, in the pneumatic trough, taking care that the neck is quite covered with water, and withdraw the thumb: water will rush into the bottle and fill the space previously occupied by the carbon dioxide. Replace the thumb on the neck of the bottle, and shake again to dissolve the last traces of carbon dioxide, and again place the neck of the bottle under water in the trough, and observe that the space occupied by water is half the total capacity of the bottle. Pour a quantity of the gas thus freed from carbon dioxide into a test tube, add a little lime water, and shake it up: no turbidity ought to be produced. Light the gas at the mouth of the test tube, observe the pale blue flame of the carbonic oxide, and after the gas is burned shake up again, and observe that the lime-water is now rendered turbid. This is because the monoxide in burning takes up oxygen from the air and produces the dioxide (CO_2).

36. *Preparation of chlorine.*[*]

When sulphuric acid acts upon a mixture of common salt and manganese dioxide, sodium sulphate, manganese sulphate, water, and chlorine gas are produced. Thus:

$$2\,NaCl + 2\,H_2SO_4 + MnO_2 = Na_2SO_4 + MnSO_4 + 2\,H_2O + Cl_2.$$

$$117 + 196 + 87 = 142 + 151 + 36 + 71.$$

Weigh out 30 grams of common salt and the same quantity of manganese dioxide, and mix them together in a mortar. Then weigh 60 grams of water, place it in an evaporating basin, and add to it cautiously 60

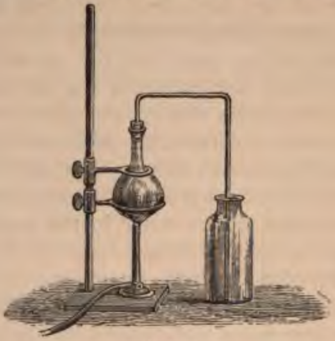

FIG. 10.

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[*] Experiments with chlorine must be made in a glass closet provided with a draught, as this gas when inhaled produces great irritation, from which serious results may ensue.

grams of strong sulphuric acid, stirring the liquid with a glass rod until it is thoroughly mixed. The liquid will become very hot, and must be allowed to cool *completely;* it is then poured into a flask (about one litre capacity), the mixture of salt and manganese dioxide is added, and the contents of the flask shaken. The flask is now provided with a conducting tube bent twice at right angles, heat is applied, and the gas collected by displacement like carbon dioxide (Fig. 10). Collect four jars of the gas, and then allow the gas to bubble through some distilled water until no more gas is dissolved. This solution, called *chlorine water*, has a yellow colour, bleaches vegetable colouring matters, and may be conveniently used instead of the gas in many reactions.

37. *Properties of chlorine.*

(*a*) 1. Chlorine unites readily with hydrogen, forming hydrochloric acid (HCl).

Place a lighted taper in a jar of chlorine, and observe that it now burns with a smoky flame; this is caused by the union of the chlorine with the hydrogen of the taper, while the carbon also present in the taper, is liberated, and causes the abundant smoke.

2. Moisten a strip of filter paper with turpentine (composed, like the taper, of carbon and hydrogen), and plunge it into a jar of chlorine: hydrochloric acid is at once formed, and the liberated carbon again appears as soot. The action is so energetic that the paper generally takes fire, owing to the heat evolved during the reaction.

(*b*) Chlorine unites readily with finely-divided metals, forming metallic chlorides.

Throw some finely-powdered antimony into a jar of chlorine: union will at once take place with evolution of light, and chloride of antimony will be formed.

(*c*) Phosphorus and chlorine unite together, forming phosphorus chloride.

Place a piece of dry phosphorus in a deflagrating spoon, and place it in a jar of chlorine. It will take fire spontaneously and continue to burn with a pale non-luminous flame, and chloride of phosphorus will be formed.

(*d*) Moist chlorine bleaches vegetable colouring matters by uniting with the hydrogen of the water; the oxygen thus liberated attacks the colouring matter and destroys it.

Place a piece of red cloth, partly moistened and partly dry, in a jar of chlorine. The dry part will remain unaltered, but the moist portion will be rapidly bleached.

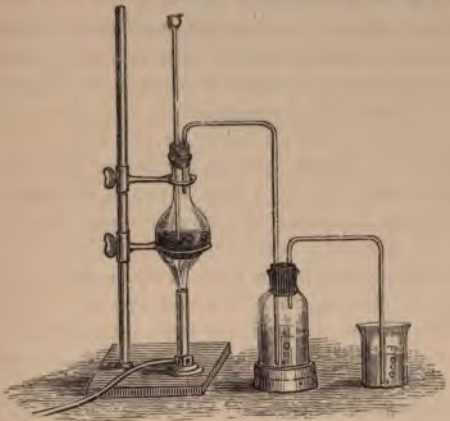
38. *Preparation of hydrochloric acid.*

When sulphuric acid and sodium chloride react on each other, hydrogen-sodium sulphate (bisulphate of soda) and hydrochloric acid are formed.

Sulphuric acid and sodium chloride yield hydrochloric acid and hydrogen-sodium sulphate:

$$H_2SO_4 + NaCl = HCl + HNaSO_4.$$
$$98 \quad + 58{\cdot}5 = 36{\cdot}5 + \quad 120.$$

Place in a flask (about a litre in capacity), provided with a funnel and conducting tube, about 30 grams of common salt. Fit the end of the conducting tube into a small bottle containing a little water, which serves to wash the gas, and the tube which passes out of this bottle conducts the purified gas into 100 c. c.* distilled water, as in the figure. When the apparatus is arranged, add by degrees about 50 grams of strong sulphuric acid. The gas will come off at first without heating; when all the acid has been added and the evolution of gas slackens, heat the flask gently on a piece of wire-gauze or on a sand bath. Observe that the water in the wash-bottle has first to be saturated, and that then the gas begins to be absorbed by the distilled water. When this last fumes strongly, remove



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* c. c. = contraction for cubic centimetres

the wash-bottle and fill a cylinder with the gas by downward displacement. (See Fig. 10.)

39. *Properties of hydrochloric acid.*

(*a*) Hydrochloric acid is extremely soluble in water.

Close the cylinder filled with hydrochloric acid gas with a glass plate, invert it, mouth downwards, in a vessel of water coloured with blue litmus, and withdraw the glass plate. Observe the rapid rise of the water inside the cylinder caused by the solution of the gas, and the change of colour from blue to red, showing the acid nature of the solution.

(*b*) Hydrochloric acid is a powerful acid.

Take some of the aqueous solution of the gas (prepared in **38**) and add a drop or two of litmus solution to it, so as to colour the solution red. Now add a solution of caustic soda, constantly stirring the mixture, and observe that a point is reached when the red colour is changed to a faint blue colour. This point denotes that all the acid has been saturated. Evaporate the solution and obtain crystals of sodium chloride (common salt).

These three substances, viz. the hydrochloric acid, the caustic soda, and the sodium chloride, are examples of three quite distinct classes of chemical compounds, called respectively, acids, bases, and salts. (For a description of these three kinds of substances see Roscoe's "Chemistry," p. 55).

(*c*) Ammonia and hydrochloric acid unite to form ammonium chloride.

Hold a glass rod dipped in ammonia solution over the HCl solution, and observe the white fumes of NH_4Cl produced. (See also **25**.)

40. *Preparation of calcium hypochlorite (or bleaching powder).*

Chlorine gas is rapidly absorbed by slaked lime, and calcium hypochlorite is formed.

Lime and chlorine yield water and bleaching powder (chloride and hypochlorite of calcium):

$$2\,Ca(HO)_2 + 4\,Cl = 2\,H_2O + (CaCl_2 + Ca(OCl)_2).$$
$$148 + 142 = 36 + 111 + 143.$$

Place a small quantity of slaked lime in a beaker, fill up with water so as to have a milky liquid containing lime in suspension, and then allow chlorine to bubble through until the solution smells strongly of it: calcium hypochlorite will be found in solution. Place a piece of red cloth in some of this solution, and subsequently in a little dilute HCl: chlorine will be liberated by the action of the acid, and the cloth will be bleached.

41. *Preparation of hypochlorous acid.*

When diluted nitric acid is added to calcium hypochlorite, hypochlorous acid and calcium nitrate are formed. Thus:

$$Ca(OCl)_2 + 2\,HNO_3 = 2\,HClO + Ca(NO_3)_2.$$
$$143 + 126 = 105 + 164.$$

Place a solution of the bleaching liquor, as prepared above, in a stoppered retort, the neck of which passes into a small flask kept cool by a stream of water. Add a few drops of *dilute* nitric acid to the contents of the retort, and boil the liquid: the distillate contains a colourless solution of hypochlorous acid, which will rapidly bleach a piece of red cloth placed in it. Observe also the peculiar smell of hypochlorous acid, and remember that a solution of chlorine in water (which likewise bleaches) has a yellow colour.

42. *Preparation of iodine.*

When sulphuric acid acts upon a mixture of potassium iodide and manganese dioxide; iodine, potassium sulphate, manganese sulphate, and water are produced. Thus:

$$\begin{array}{l} 2KI + MnO_2 + 2H_2SO_4 = I_2 + K_2SO_4 + MnSO_4 + 2H_2O \\ 332{\cdot}2 + \ 87 \ + \ 196 \ = 254 + 174{\cdot}2 + \ 151 \ + \ 36. \end{array}$$

Place in a retort (provided with a receiver kept cold by a stream of water) a few grams of potassium iodide and a little manganese dioxide, add water and a little *dilute* sulphuric acid, and heat gently. Observe the violet vapours filling the retort as soon as the iodine is given off, and the grey deposit of iodine on the neck of the retort and in the receiver. If a large quantity forms in the retort neck, heat it gently, so as to obtain as much as possible in the receiver.

43. *Properties of iodine.*

(*a*) Iodine is very sparingly soluble in water, more so in alcohol, and very soluble in a solution of any alkaline iodide.

Separate the iodine obtained above, from any liquid which has distilled over, and divide it into three small portions and one large portion. Add to one of the small portions some water, to another some alcohol, and to the third a solution of potassium iodide, and observe the colours varying from pale to dark brown, which the solutions possess.

(*b*) Free iodine forms with starch a compound called iodide of starch, which has a dark blue colour.

Powder a piece of starch (about the size of a pea) in a mortar, stir it up with about 25 c. c. of cold water, and then heat the mixture (preferably in an evaporating basin) till it boils. A thin, clear solution of starch is thus obtained; add a portion of it to about a quarter of a litre of water, and then a few drops of one of the solutions of iodine, and observe the deep blue colour which the liquid assumes. Heat a little of this blue liquid in a test tube, and observe that the colour disappears; allow it to cool, and observe the reappearance of the colour.

44. *Preparation of sodium iodide.*

Iodine is dissolved by a solution of caustic soda, and sodium iodide (NaI) and iodate ($NaIO_3$) are produced.

$$\begin{array}{ccccccccc} 6\,I & + & 6\,NaHO & = & 5\,NaI & + & NaIO_3 & + & 3\,H_2O. \\ 762 & + & 240 & = & 750 & + & 198 & + & 54. \end{array}$$

Place the larger portion of iodine prepared as above in an evaporating basin, add water and caustic soda drop by drop until the solution becomes nearly colourless, and evaporate carefully to dryness, and ignite to convert the sodium iodate into iodide. ($NaIO_3 = NaI + O_3$). A white salt is obtained, which is sodium iodide.

(*a*) Dissolve it in water and add to it about ¼ litre of water, then add a little of the starch solution, and observe that no blue colour is produced as in the case of *free* iodine. Now add a drop of chlorine water: this will liberate the iodine and the blue colour will be produced; add more chlorine water, and the colour will disappear, owing to the formation of a chloride of iodine which does not colour starch blue.

(*b*) Add a few drops of the sodium iodide solution to some distilled water, then solution of silver nitrate, and observe the pale yellow precipitate of insoluble silver iodide which is produced. Sodium iodide and silver nitrate yield sodium nitrate and silver iodide:

$$NaI + AgNO_3 = NaNO_3 + AgI.$$
$$150 + 170 = 85 + 235.$$

45. *Preparation of bromine.*

When sulphuric acid acts upon a mixture of potassium bromide and manganese dioxide, potassium sulphate, manganese sulphate, bromine, and water are produced. Thus:

$$2\,KBr + MnO_2 + 2\,H_2SO_4 = K_2SO_4 + MnSO_4 + Br_2 + 2\,H_2O.$$
$$238{\cdot}2 + 87 + 196 = 174{\cdot}2 + 151 + 160 + 36.$$

Proceed exactly as in the preparation of iodine (**42**), but substitute potassium bromide for potassium iodide, and keep the receiver very cold. A dark-coloured heavy liquid will be obtained in the receiver: this is bromine. Observe that it is heavier than the water, which always distils over with it, and that it possesses a powerful irritating smell. Observe also that it dissolves in water, forming a red-coloured solution, which possesses bleaching properties.

46. *Preparation of sodium bromide.*

Bromine is dissolved by a solution of caustic soda and sodium bromide (NaBr), sodium bromate ($NaBrO_3$) and water are produced. Thus :

$$6\,Br + 6\,NaHO = 5\,NaBr + NaBrO_3 + 3\,H_2O.$$
$$480 + 240 = 515 + 151 + 54.$$

Proceed as in the preparation of sodium iodide (**44**), substituting bromine for iodine. Dissolve the salt obtained in water, and observe that the solution is colourless ; add chlorine water and observe the yellow colour produced by the liberation of bromine.

47. *Preparation and properties of hydrofluoric acid.*

When sulphuric acid acts upon calcium fluoride (fluor-spar), calcium sulphate and hydrofluoric acid are produced. Thus :

$$CaF_2 + H_2SO_4 = CaSO_4 + 2\,HF.$$
$$78 + 98 = 136 + 40.$$

This gas cannot be collected in glass vessels as it combines with the silica of glass, forming silicon tetrafluoride, SiF_4. This property may be observed by covering a glass plate with a thin coating of bees wax, scratching away the wax at certain points, and then exposing the plate to the action of the gas. Place some powdered fluor-spar in a small lead or platinum dish, pour over it some strong sulphuric acid, and heat gently. Observe the fumes of hydrofluoric acid which come off, and then place the waxed glass plate across the dish so as to be exposed to the gas, taking care that the heat applied is not sufficient to melt the wax. Remove the plate after a few minutes, warm it to soften the wax, which may then be rubbed off, and observe that where the wax was scratched the glass is etched, while the part protected by wax has not been attacked.

Avoid breathing the fumes, as the gas is very irritating to the lungs, and acts powerfully on the skin, producing painful wounds.

48 *Preparation of sulphur dioxide.*

When sulphuric acid acts upon copper, copper sulphate, sulphur dioxide, and water are produced. Thus:

$$Cu + 2\,H_2SO_4 = CuSO_4 + SO_2 + 2\,H_2O.$$
$$63{\cdot}5 + 196 = 159{\cdot}5 + 64 + 36.$$

Place some copper turnings in a flask of about half a litre in capacity, provided with a funnel and conducting tube bent twice at right angles. Pour enough strong sulphuric acid down the funnel tube to cover the copper, and apply heat, collecting the gas, which is heavier than air, by downward displacement (see Fig. 10). Fill two jars with the gas, and then allow it to pass, first into a test tube containing nitric acid, and then into one containing a solution of potassium chromate. Observe in the first case the brown vapours of oxides of nitrogen which are given off, and in the second the change of colour from yellow to green. In both cases the sulphur dioxide has acted as a reducing agent, reducing the nitric acid to a lower stage of oxidation, and the potassium chromate to a chromium salt of a green colour; while in each case the sulphur dioxide is oxidized to sulphuric acid, as may be seen by adding barium chloride, which will precipitate white barium sulphate, insoluble in hydrochloric acid.

49. *Properties of sulphur dioxide.*

(*a*) Sulphur dioxide does not support combustion, and reddens litmus solution.

Place a lighted taper in a jar of the gas and observe that it is at once extinguished. Then add a solution of litmus, and observe the bright red colour produced.

(*b*) Sulphur dioxide is extremely soluble in water.

Invert a jar of the gas in the pneumatic trough, and observe the rapid rise of water inside the jar, produced by the absorption of the gas.

50. *Preparation of sulphuretted hydrogen.*

When sulphuric acid acts upon ferrous sulphide, ferrous sulphate and sulphuretted hydrogen are produced. Thus:

$$\begin{array}{ccccc} FeS + H_2SO_4 & = & FeSO_4 + H_2S. \\ 88 + 98 & = & 152 + 34. \end{array}$$

Place a few pieces of ferrous sulphide in a small flask provided with a funnel and conducting tube, the latter of which passes air-tight into a small flask containing water (to collect any impurities which may pass over), and having a second tube fitted into it and bent so as to pass into a flask containing water (see figure). Now cover the ferrous sulphide with a layer of water, and add a few drops of strong sulphuric acid: observe the effervescence which soon begins, and the disagreeable and characteristic smell which the water in the flask soon possesses from the solution of the gas in it. When the water smells strongly, remove the flask;* then decant off the acid from the ferrous sulphide, wash it two or three times with water, retaining it in the flask so that it may be used again by simply adding fresh acid.

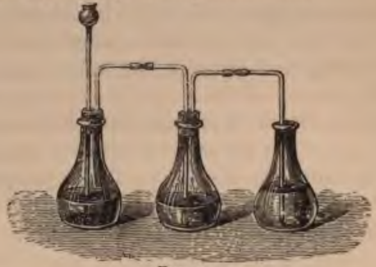

FIG. 12.

51. *Properties of sulphurretted hydrogen.*

Sulphurretted hydrogen precipitates the solutions of salts of certain metals in an acid solution, others in an alkaline solution, and does not, under any circumstances, precipitate the remainder; thus, solutions of copper salts are precipitated in an acid solution, solutions of iron salts in an alkaline one, and solutions of sodium salts are not precipitated at all. Thus:

Copper sulphate and sulphuretted hydrogen yield copper sulphide and sulphuric acid:

$$CuSO_4 + H_2S = CuS + H_2SO_4.$$
$$159{\cdot}5 + 34 = 95{\cdot}5 + 98.$$

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* The solution decomposes on standing. It is best preserved in a corked bottle, kept inverted in a vessel of water.

Iron sulphate and potash and sulphuretted hydrogen yield iron sulphide, potassium sulphate, and water.

$$FeSO_4 + 2\,KHO + H_2S = FeS + K_2SO_4 + 2\,H_2O.$$
$$152 + 112{\cdot}2 + 34 = 88 + 174{\cdot}2 + 36.$$

Place in a test glass a solution of copper sulphate, in another a solution of iron sulphate, and in a third a solution of sodium chloride; to each add a few drops of hydrochloric acid, and then a little sulphuretted hydrogen water. Observe the black precipitate of copper sulphide in the first glass, and no precipitate in the other two glasses. To each of these add a little potash solution, and observe the black precipitate of ferrous sulphide in the one case, and the absence of a precipitate in the other.

52. *Preparation of sodium hydrate (caustic soda).*

When caustic lime is added to a solution of sodium bicarbonate, caustic soda and calcium carbonate are produced. Thus:

$$CaO + NaHCO_3 = NaHO + CaCO_3.$$
$$56 + 84 = 40 + 100.$$

Dissolve about 40 grams of sodium bicarbonate in about half a litre of hot water. Then weigh out 30 grams of quick lime, slake it with water, and when it is thoroughly slaked, stir it up with more water so as to obtain a milky fluid having lime in suspension. Add this to the hot solution of sodium bicarbonate, and boil for a few minutes. Withdraw the lamp, allow the precipitate to subside, and observe if a small portion of the clear liquid effervesce when hydrochloric acid is added to it. If so, there is still some sodium bicarbonate unconverted into caustic soda, and more lime must, therefore, be added. If, on the other hand, no effervescence occurs, the decomposition is complete, and the clear liquid is then evaporated in a clean iron or silver dish to dryness. The resulting white substance is sodium hydrate ($NaHO$) or caustic soda.

53. *Properties of sodium hydrate.*

Sodium hydrate is a powerful alkali, and turns red litmus solution blue. When hydrochloric acid is added to it, it is neutralized, and sodium chloride (common salt) formed. (See **39**)

Sodium hydrate and hydrochloric acid yield sodium chloride and water:

$$\begin{array}{ccccccc} NaHO & + & HCl & = & NaCl & + & H_2O. \\ 40 & + & 36{\cdot}5 & = & 58{\cdot}5 & + & 18. \end{array}$$

Dissolve some of the caustic soda obtained in **52**, in water, and add to it a solution of reddened litmus, and observe the change in colour from red to blue. Dissolve a second portion in water, and add to it hydrochloric acid by degrees, until a drop of the liquid taken out on a glass rod ceases to colour litmus paper blue. On evaporating the liquid thus obtained to a small bulk, sodium chloride will separate out.

PART II.

BLOW-PIPE ANALYSIS.—PRELIMINARY EXAMINATION.

54. *Blow-pipe reactions.*

How to use the blow-pipe.*

Close the holes at the foot of the Bunsen lamp (Fig. 13) so as to exclude air, and thus obtain a luminous flame. Now place the nozzle of the blow-pipe in the centre of the flame, and blow gently through the tube: observe that the flame produced is blue and corresponds to the non-luminous flame of the Bunsen lamp. This is the *oxidizing* or *outer* flame of the blow-pipe.

Now hold the nozzle of the blow-pipe *just outside* the luminous gas flame, and blow gently: observe that the flame is partly yellow. This is the *reducing* or *inner* flame of the blow-pipe (Fig. 14).

The oxidizing flame is used when a substance has to be oxidized, the reducing flame when a body has to be reduced, *e.g.* from a salt to the metallic state.

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* The student should be shown, once for all, the different uses of the blow-pipe, and then be allowed to practise on several different substances Unless when otherwise expressed the substances used must be dry.

EXAMPLES.

1. *Reduction.*—Mix together in a mortar equal small quantities of dry sodium bicarbonate and silver nitrate. Place a portion in a little hollow scooped out of a sound piece of charcoal, and heat in the *reducing* flame of the blow-pipe. Observe the bright metallic bead of silver obtained, dissolve it in nitric acid, and precipitate it as chloride with a few drops of HCl.

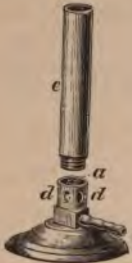

FIG. 13.

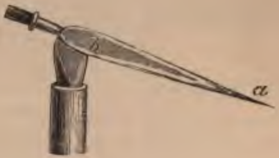

FIG. 14.

2. *Oxidation.*—(*a*) Make a small loop on the end of a piece of platinum wire, heat it, and dip it while hot in some sodium bicarbonate, so as to cause a small quantity to adhere to the wire; now heat it with the blow-pipe flame until it is fused.* Then place on it a minute quantity of any manganese compound, and heat again in the *oxidizing* flame of the blow-pipe; by

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* In a similar way, beads are also made with borax or microcosmic salt, instead of sodium bicarbonate.

this means sodium manganate is formed, which colours the bead bright green.

(*b*) Heat a small portion of metallic lead on a piece of charcoal in the oxidizing blow-pipe flame. Observe the yellow incrustation on the charcoal produced by the *oxidation* of the lead to litharge (PbO).

55. *Blow-pipe reactions for the commonly occurring metals.**

(*a*) Compounds reduced to metal when heated with $NaHCO_3$ on charcoal in reducing flame :

$$\underbrace{\textbf{Silver.}\quad\textbf{Lead.}}_{\text{Malleable beads.}}\qquad\qquad\underbrace{\textbf{Bismuth.}\quad\textbf{Antimony.}}_{\text{Brittle beads.}}$$

(*b*) Compounds reduced to metal when heated with a mixture of KCN and $NaHCO_3$ in reducing flame :

Tin.	**Copper.**
White.	Red.

(*c*) Compounds reduced to metal when heated on charcoal with reducing agents, but which at once volatilize:

Mercury. **Arsenic.**

(*d*) Compounds reduced to magnetic metallic powders when heated on charcoal with reducing agents :

Iron. **Nickel.** **Cobalt.**

(*e*) Compounds reduced to metal when heated on charcoal with reducing agents, but which are at once converted into oxides :

Cadmium.	**Zinc**
Oxide is brown.	Oxide is white.

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* These reactions must be carried out *in the order indicated* if they are applied to the examination of an unknown compound, since a metallic salt which is reduced by $NaHCO_3$ alone, is reduced *a fortiori* by a mixture of KCN and $NaHCO_3$.

(*f*) Compounds which, after being heated on charcoal, then moistened with solution of $CoCl_2$, and heated again, yield characteristic colours :

Zinc.	**Aluminium.**	**Magnesium.**
Green.	Blue.	Pink.

(*g*) Compounds which when fused in a borax bead impart to it a characteristic colour :

Iron.	**Cobalt.**	**Nickel.**	**Manganese.**	**Chromium.**	**Copper.**
Yellow.	Blue.	Reddish yellow.	Amethyst.	Green.	Blue.

(*h*) Compounds which impart a characteristic colour to any non-luminous flame :

Barium.	**Strontium.**	**Calcium.**	**Potassium.**	**Sodium.**
Green.	Crimson.	Red.	Violet.	Yellow.

56. Having found out *approximately* by these reactions what the substance is, proceed to apply the following

Confirmatory Tests.

(a*) Silver bead soluble in HNO_3. Solution yields with HCl white curdy precipitate of AgCl.

Lead bead soluble in HNO_3. Solution yields with dilute H_2SO_4 heavy white precipitate of $PbSO_4$.

Bismuth bead soluble in HNO_3. Solution evaporated with HCl yields with H_2S *black* precipitate of Bi_2S_3.

Antimony bead soluble in HNO_3. Solution evaporated with HCl yields with H_2S *orange* precipitate of Sb_2S_3.

(*b*) Tin bead soluble in HNO_3. Solution on evaporation yields white precipitate of SnO_2.

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* The letters correspond with those in the preceding paragraphs.

Copper bead soluble in HNO_3. Solution, on addition of $(NH_4)HO$ in excess, yields deep blue solution.

(*c*) Mercury compounds, when heated in a small bulb-tube with $NaHCO_3$, yield the metal in minute globules.

Arsenic compounds, when heated in a small bulb-tube with $KCN + NaHCO_3$ yield the metal as a shining mirror.

(*d*) Iron powder soluble in $HNO_3 + HCl$ yields yellow solution which gives a deep blue coloration with $K_4Fe(CN)_6$.

Cobalt powder soluble in HNO_3 yields red solution, which gives a blue bead when fused with borax.

Nickel powder soluble in HNO_3 yields green solution, which gives a reddish yellow bead when fused with borax.

(*e*) Cadmium oxide distinguished by its brown colour on the charcoal.

Zinc oxide distinguished by its yellow colour while hot, turning white when cold. (See also under *f*.)

(*f*) Zinc compounds, a green residue on charcoal when moistened with $CoCl_2$ and re-heated. (See also under *e*.)

Aluminium compounds, a blue residue on charcoal when moistened with $CoCl_2$ and re-heated.

Magnesium compounds, a pink residue on charcoal when moistened with $CoCl_2$ and re-heated.

Best seen when cold.

(*g*) Iron borax bead, reddish yellow when hot, pale yellow on cooling in oxidizing flame.

Iron borax bead, light green in reducing flame.

Cobalt borax bead, deep blue in either oxidizing or reducing flame.

Nickel borax bead, reddish yellow when hot, paler on cooling, and finally nearly colourless in oxidizing flame.

Nickel borax bead, grey when heated in the reducing flame.

Manganese borax bead, amethyst-coloured in oxidizing flame, colour disappears in reducing flame.

Chromium borax bead, green in either oxidizing or reducing flame.

Copper borax bead, blue or greenish-blue in the oxidizing flame, becomes colourless in the reducing flame. (See also under *b*.)

(*h*) Barium salts colour the non-luminous gas flame *pale green*.

Strontium salts colour the non-luminous gas flame *bright crimson*.

Calcium salts colour the non-luminous gas flame *dull red*.

Potassium salts colour the non-luminous gas flame *violet*.

Sodium salts colour the non-luminous gas flame *yellow*.

Best seen after moistening the salt with HCl.

BUNSEN'S FLAME REACTIONS.

57.—The flame of an ordinary Bunsen lamp serves for nearly all the reactions which can be performed by the mouth blow-pipe. It is most convenient to use a lamp in which the admission of air can be regulated, and in which the flame is kept steady by a conical chimney supported from the tube of the lamp (see Fig. 15). Adjust the brass cap covering the holes *d d*, Fig. 13, so as to obtain a small luminous point at η, Fig. 15, and then notice the following zones of flame, and the purposes to which they are best suited.

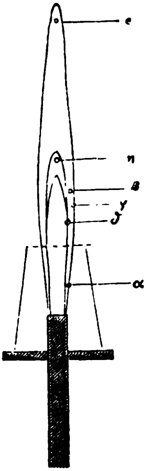

FIG. 15.

a. Temperature low. Suitable for observing flame colorations of volatile substances.

β. Highest temperature. Suitable for fusions at high temperatures.

γ. *The lower oxidizing flame.* Suitable for oxidation of substances in borax or other beads.

δ. *The lower reducing flame.* Suitable for reductions on charcoal, and in fused borax or other beads.

ϵ. *The upper oxidizing flame* (obtained by admitting the maximum of air). Suitable for oxidation at lower temperatures than are found at β and γ.

η. *The upper reducing flame.* Suitable for reductions; possesses greater reducing power than δ

Metallic films.—The more volatile metals, such as arsenic, mercury, and zinc, are reduced from their compounds when these are heated on an asbestos thread in the upper reducing flame (η). If a small porcelain basin, filled with cold water, be held just above the substance to be examined, the volatilized metal condenses on the cold basin as a metallic film.

Example.—Place a minute quantity of any arsenic compound on a thread of asbestos. Hold this in one hand, and in the other a small porcelain basin filled with cold water. Now place the basin just above the upper reducing flame, and then the asbestos thread immediately below. In a few seconds the reduction will be complete; remove the basin and observe the brown film of metallic arsenic. Moisten the film with cold dilute nitric acid, and observe that it is scarcely soluble; moisten now with solution of sodium hypochlorite, and observe its instant solubility.

Metallic beads.—The less volatile metals may be obtained as beads, when their compounds are heated with sodium carbonate on a small charcoal rod held in the lower reducing flame (δ).

Example.—Hold a crystal of sodium carbonate in the lamp flame for a few seconds till it begins to fuse; then rub the fused salt thus obtained over a common wooden lucifer match, and burn this until it is converted into a rod of charcoal. Then allow a single drop of the fused sodium carbonate to fall on the palm of the hand, mix this intimately by means of a pen-knife with a small quantity of silver nitrate, and then place a very small portion of the mixture on the point of the rod of charcoal. Allow the mixture to melt in the lower oxidizing flame (γ), and then push the charcoal splinter into the lower reducing flame (δ). When the reduction is completed, remove the splinter and examine the point with a lens. Minute beads of fused silver will be seen, which may be further examined by breaking off the end of the splinter, and crushing it, along with a few drops of water in a small agate mortar. Pour off the water (which will carry the charcoal with it), and examine the metal thus obtained in the same way as the bead obtained by the mouth blow-pipe (**56**, *a*).

58. Compounds of the following metals form metallic films:—ANTIMONY, ARSENIC, BISMUTH, MERCURY, THALLIUM, CADMIUM, ZINC, INDIUM, and LEAD. They may be further distinguished by the following tests:—

Antimony.	Black film, thin part brown	Scarcely soluble in cold dilute HNO_3.
Arsenic.	,, ,, ,,	
Bismuth.	Black film, thin part brown	With difficulty soluble in cold dilute HNO_3.
Mercury.	Grey non-coherent thin film	
Thallium.	Black film, thin part brown	
Cadmium.	Black film, thin part brown	Instantly soluble in cold dilute HNO_3.
Zinc.	,, ,, ,,	
Indium.	,, ,, ,,	
Lead.	,, ,, ,,	

Compounds of the following metals give no film, but are reduced to metal on charcoal splinter:—COPPER, TIN, SILVER, GOLD, PLATINUM, IRON, NICKEL, and COBALT. They may be further distinguished as follows:—

Copper.	Red bead, soluble in HNO_3	Fusible to metallic beads.
Tin.	White bead ,, ,,	
Silver.	,, ,, ,,	
Gold.	Yellow bead, insoluble ,,	
Platinum.	Non-magnetic powder	Not fusible to beads, but obtained as metallic powders.
Iron.	Magnetic powder	
Nickel.	,, ,,	
Cobalt.	,, ,,	

59. Additional tests for the compounds of the following metals:—

Antimony.—On asbestos thread in upper reducing flame, pale green coloration, unaccompanied by smell. Reduced on charcoal splinter, yields white brittle metallic beads.

Arsenic.—On asbestos thread in upper reducing flame, pale blue coloration, and characteristic smell (garlic). Reduced on charcoal splinter, yields no metallic bead.

Bismuth.—Reduced on charcoal splinter, yields shining yellowish brittle splinters of metal. Dissolve in HNO_3, add $SnCl_2$, and $NaHO$, yields black precipitate of $Bi_2 O_2$.

Mercury.—Mixed with dry sodium carbonate and potassium nitrate, in a small thin test-tube (5 millimetres wide and 15 millimetres long), and held in the flame (by a platinum wire coiled round it) just below a small porcelain basin filled with water, yields grey film, which, on rubbing with a piece of filter paper, is collected into small globules. If the quantity of mercury be large, globules form at once.

Thallium.—Flame coloration bright green. Reduced on charcoal splinter, yields white ductile bead, which quickly oxidizes, and is acted on by HCl with difficulty.

Cadmium.—Reduced on charcoal splinter imperfectly to a white ductile bead.

Zinc.—On asbestos thread in upper oxidizing flame, yields white film of ZnO on the porcelain basin. Moisten a square centimetre of filter paper with HNO_3, and rub it over the surface of the basin so as to dissolve the oxide film; roll this up and place it in a coil of thin platinum wire. Now burn the paper in the upper oxidizing flame at as low a temperature as possible, and observe that the colour of the ash is yellow, while hot, and white on cooling. Moisten this ash with a drop of a very dilute solution of cobalt, heat in the lamp flame, and observe the green colour produced.

Indium.—Flame coloration, intense indigo colour. Reduced on charcoal splinter with difficulty to silver-white ductile globules, slowly soluble in HCl.

Lead.—Reduced on charcoal splinter, yields soft ductile metallic beads, soluble in HNO_3. Add H_2SO_4, yields white precipitate of $PbSO_4$.

Copper.—Reduced on charcoal splinter, yields ductile metallic beads of a red colour. Dissolve in HNO_3 and add $K_4Fe(CN)_6$, yields chocolate coloured precipitate of $Cu_2Fe(CN)_6$.

Fuse a small quantity of borax on a straight piece of platinum wire (of the thickness of a horsehair), and, having obtained a clear bead, add a trace of any copper compound, and observe the blue bead obtained, which does not alter in the lower reducing flame. Add a trace of tin or any tin salt, and heat in the lower reducing flame; observe the change of colour to red, owing to the formation of Cu_2O.

Tin.—Obtain a borax bead coloured faintly blue by copper, and add the tin compound; proceed just as described under copper. The change of colour from blue to red indicates presence of tin.

Silver.—Reduced on charcoal splinter, yields white ductile beads. Dissolve in HNO_3 and add HCl, yields white curdy precipitate of AgCl.

Gold.—Reduced on charcoal splinter, yields yellow very malleable beads. Dissolve in *aqua regia*, absorb the yellow solution on filter paper, and moisten with $SnCl_2$, yields purple of Cassius.

Platinum.—Reduced on charcoal splinter yields a grey spongy mass, which becomes lustrous when rubbed in the mortar. Observe insolubility in HCl and in HNO_3, and solubility in *aqua regia*, forming a light yellow solution.

Iron.—Reduced on charcoal splinter, yields no bead, but minute metallic particles. Crush the end of the splinter in an agate mortar with a little water, and stir gently with a magnetized knife-blade. The finely divided metal will adhere to the knife. Rub this off on filter-paper, dissolve in *aqua regia* and add $K_4Fe(CN)_6$: observe the blue coloration from formation of Prussian blue.

Borax bead: In the oxidizing flame when hot, yellow to brownish red.
" " In the oxidizing flame when cold, yellow to brownish yellow.
" " In the reducing flame, bottle green.

Nickel.—Reduced on charcoal splinter, yields white lustrous ductile particles, which form a brush on the point of the magnetized knife-blade. Rub these on paper and dissolve in HNO_3, and observe the green colour produced.

Borax bead: In the oxidizing flame, greyish brown or dirty violet.

Upper reducing flame, grey from reduced nickel which often collects to a spongy mass, leaving the bead colourless.

Cobalt.—Reduced on charcoal splinter, yields white lustrous ductile particles, which adhere to the magnetized knife-blade. Rub these on paper and moisten with HNO_3: observe the red colour, which changes to green on addition of HCl and drying; moisten with water, and observe the disappearance of the colour.

Borax bead: In the oxidizing flame, bright blue, unaltered in the lower reducing flame.

60. The following metals are most easily recognized as compounds:—CHROMIUM, MANGANESE, URANIUM.

Chromium.—Heated on platinum foil with Na_2CO_3 and with repeated additions of KNO_3, yields a yellow mass soluble in water to a yellow solution.

Manganese.—Borax bead amethyst-coloured in the oxidizing flame, colourless bead in the reducing flame. Heated with Na_2CO_3 and KNO_3, yields a green bead, soluble in water to a green solution, which turns red on addition of acetic acid.

Uranium.—Borax bead yellow, in the oxidizing flame, which becomes green in the reducing flame, especially on addition of $SnCl_2$.

The following tests for phosphorus and sulphur compounds are exceedingly delicate:—

Phosphorus.—Ignite the sample, and then powder finely and place in a small test tube about the thickness of a straw, along with a piece of magnesium wire about five millimetres long, which should be covered by the powder. Now heat, and observe the incandescence caused by the formation of magnesium phosphide. Moisten the residue, and observe the highly characteristic smell of phosphuretted hydrogen.

Sulphur. — Reduced on charcoal splinter with Na_2CO_3, yields Na_2S. Break off the charcoal point, place it on a silver coin, and moisten with a drop of water. The silver is at once blackened, owing to the formation of silver sulphide.

61.—PRELIMINARY EXAMINATION OF SINGLE SALTS.

Before proceeding to the systematic analysis of single salts or mixtures, it is always advantageous to subject substances to a *preliminary examination*, in order to ascertain the probable nature of the substance. The tables A and B, which follow, are adapted for the detection of single salts, either soluble or insoluble.

TABLE A.

PRELIMINARY EXAMINATION OF SINGLE SALTS CONTAINING ONE ACID AND ONE BASE.

a. EXAMINATION FOR ACID.*

Heat the substance in a dry tube, and observe whether—

(*a*) Water is given off. If so, test its reaction with litmus paper.

Acid reaction indicates **Sulphites** (105),† **Chlorides** (111), &c.

Alkaline reaction indicates **Ammonium Salts** (91).

(*b*) A sublimate forms—

Yellow (or in red globules)	indicates	**Sulphur** (60).
White	,,	**Ammonium Salts** (91), Selenium, **Mercury** (64 or 67), **Antimony** (74), **Arsenic** (75). Tellurium
Metallic mirror	,,	**Mercury** (64 or 67), **Arsenic** (75). [illegible]

(*c*) A gas is evolved—

Oxygen	indicates	**Chlorates** (119), **Nitrates** (23), **Peroxides.**
Carbon monoxide	,,	**Oxalates** (100).
Nitrogen tetroxide	,,	**Nitrates** (23).
Ammonia	,,	**Ammonium Salts** (91).
Carbon dioxide	,,	**Carbonates** (102).

(*d*) The substance alters in colour. To black, indicates **Organic matter,** to yellow (while hot), indicates **Zinc Oxide** or **Carbonate,** to brown, indicates **Cadmium Carbonate.**

Take a fresh portion of the substance, add HCl, and observe whether—

(*e*) A gas is evolved with effervescence.

Smelling like burning sulphur	indicates	**Sulphites** (105) or **Thiosulphates** (106).
,, ,, rotten eggs	,,	**Sulphides** (117).
,, ,, bitter almonds	,,	**Cyanides** (114).
,, of chlorine (on heating)	,,	**Peroxides, Chromates** (110), **Hypochlorites** (115)
Rendering lime water turbid	,,	**Carbonates** (102).

(*f*) Try if the substance is soluble in water; if so, add $BaCl_2$ solution to a portion of the solution, and observe if a precipitate form.

White and insoluble in HCl indicates **Sulphates** (96).

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* Certain substances, not acids (*e.g.* mercury, sulphur, ammonia), are for convenience included here.

† These numbers refer to the paragraphs where the reactions of the acids and bases are to be found.

White and soluble in HCl indicates **Phosphates** (98), **Silicates** (103), **Oxalates** (100), **Borates** (99), and **Fluorides** (101). Also **Carbonates** and **Sulphites** (see *e*).

If $BaCl_2$ has given no precipitate, add $AgNO_3$ to another portion of the solution, and observe if a precipitate form.

White and insoluble in HNO_3 indicates **Chlorides** (111), or **Cyanides** (114) (see *e*).
Yellowish-white and insoluble in HNO_3 indicates **Bromides** (112) and **Iodides** (113).
Yellow and soluble in HNO_3 indicates **Phosphates** (98).
Black indicates **Sulphides** (117) (see *e*).

(*g*) If neither water nor HCl has dissolved the substance, try nitric acid; and if this does not dissolve it, try *aqua regia;* and if this does not dissolve it, examine the substance according to the methods described in Table B, page 58.

NOTE.—Use as *little acid as possible* to dissolve the substance. If nitric acid or *aqua regia* has been used, evaporate the solution to dryness with HCl before proceeding to examine for the base.

β. EXAMINATION FOR BASE.

A solution having been obtained, observe—

(*h*) If HCl produce a precipitate, it indicates—
Silver (63), **Mercurous Salts** (64), or **Lead** (65).

(*i*) If HCl + H_2S produce a precipitate, it indicates—
If black, **Mercuric Salts** (67), **Lead** (65), **Bismuth** (69), or **Copper** (70).
If yellow, **Cadmium** (71), **Arsenic** (75), or **Stannic Salts** (73).
If orange, **Antimony** (74).
If brown, **Stannous Salts** (73).

(*j*) If $(NH_4)HO + NH_4Cl + (NH_4)_2S$ produce a precipitate, it indicates—
If black, **Iron** (77), **Nickel** (78), or **Cobalt** (79).
If white, **Zinc** (80), or **Aluminium** (81).
If flesh-coloured, **Manganese** (82).
If green, **Chromium** (83). (All Chromium compounds are coloured.)

(*k*) If $(NH_4)HO + NH_4Cl + (NH_4)_2CO_3$ produce a precipitate, it indicates—
Barium (85) (tinges flame green). The colours are best
Strontium (86) (,, ,, crimson). seen after moistening
Or **Calcium** (87) (,, ,, dull red). the salts with HCl.

(*l*) If not precipitated by the above reagents, it indicates—
Magnesium (89), precipitated by $Na_2HPO_4 + (NH_4)HO$ (white).
Potassium (90) (tinges flame violet).
Sodium (92) (,, ,, yellow).
Or **Ammonium Salts** (91) heated with NaHO give smell of NH_3.

TABLE B.

EXAMINATION OF INSOLUBLE SUBSTANCES.

The following substances are, under certain circumstances, insoluble in acids, and must be examined specially:—**Silica, Silicates, Aluminia, Aluminates, Oxides of Antimony, Chromium,** and **Tin, Chrome Iron Ore, Sulphates of Barium, Strontium,** and **Lead, certain Fluorides** (*e.g.* of Calcium), **certain Sulphides** (*e.g.* of Lead), the **Chloride, Bromide, and Iodide of Silver, Carbon, and Sulphur.**

Heat the substance in a dry tube, and observe whether—

(*a*) It fuses and volatilizes completely. **Sulphur** (smells of SO_2).

(*b*) It fuses, but does not volatilize. **Chloride, Bromide,** or **Iodide of Silver.** (Yields metallic silver on fusing on charcoal with Na_2CO_3).

(*c*) It is infusible, but disappears on heating. **Carbon** (Deflagrates when heated with KNO_3).

(*d*) It is infusible, but darkened in colour while hot, regaining its colour on cooling. **Tin Dioxide** and **Antimony Pentoxide.** (Confirm by blow-pipe test—tin bead is malleable; antimony bead brittle.)

Observe whether—

(*e*) It yields a green bead with borax or microcosmic salt. **Chromium Oxide** or **Chrome Iron Ore.**

(*f*) It swims undissolved in a bead of microcosmic salt. **Silica** and **Silicates.** (Fuse with four times its weight of a mixture of K_2CO_3 and Na_2CO_3. Allow to cool, dissolve in water, add HCl, and evaporate to dryness. Silica will separate out as a gelatinous mass.)

(*g*) It yields a colourless bead, with microcosmic salt. **Alumina.** (Heated on charcoal and moistened with $Co(NO_3)_2$ and re-heated, it yields a blue infusible mass.)

(*h*) It is white and infusible, but quite unaltered by heating.

Lead Sulphate yields, when heated with Na_2CO_3 in blow-pipe reducing flame, malleable metallic bead. (See also *i.*)

change color in closed tube. ZnO white – yellow becom white on cooling. SnO white – yellowish brownish di pale yellow on cooling. PbO (fusible) white – brownish yellow when cold. Bi O white – orange yellow in cold orange pale yellow when cold. HgO (volatile) red – black in cold. FeO red – black red when cold.

Barium Sulphate, fused with Na_2CO_3 yields $BaCO_3$. Boil the fused mass with water, filter and wash; the residue dissolved in HCl yields $BaCl_2$ (flame coloration, green), precipitated by $SrSO_4$ solution.

Strontium Sulphate, fused with Na_2CO_3 yields $SrCO_3$. Boil the fused mass with water, filter and wash; the residue dissolved in HCl yields $SrCl_2$ (flame coloration, crimson), precipitated by $CaSO_4$ solution.

Calcium Fluoride, heated with H_2SO_4, yields HF, which etches glass.

(*i*) It is black and infusible, and yields a malleable metallic bead when fused with Na_2CO_3 in the blow-pipe flame. **Lead Sulphide.** (Bead leaves black mark on paper; and, when dissolved in HNO_3, gives a white precipitate on addition of H_2SO_4.

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* The action of strong sulphuric acid often affords a valuable indication of the nature of salts, both soluble and insoluble. In addition to observing the action of hydrochloric acid (*e*, Table A), it is always advisable to try the action of strong sulphuric acid. Thus, evolution of—

	Sulphur Dioxide	indicates	**Sulphites** (105). **Thiosulphates** (106).
	Sulphuretted Hydrogen		**Sulphides** (117).
	Hydrocyanic Acid	,,	**Cyanides** (114)
	Oxygen	,,	**Peroxides.** **Chromates** (110). **Permanganates.**
	Carbon Dioxide	,,	**Carbonates** (102).
	Carbon Monoxide	,,	**Oxalates** (100). **Formates** (129.) **Ferrocyanides** (126).
	Chlorine	,,	**Hypochlorites** (115).
	Chlorine Tetroxide	,,	**Chlorates** (119).
Fuming Gases	Hydrochloric Acid	,,	**Chlorides** (111).
	Hydrobromic Acid (+ Br. vapour)	,,	**Bromides** (112).
	Hydriodic Acid (+ I vapour)	,,	**Iodides** (113).
	Hydrofluoric Acid	,,	**Fluorides** (101).
	Nitric Acid	,,	**Nitrates** (23).
	Oxides of Nitrogen	,,	**Nitrites** (116).
	Acetic Acid	,,	**Acetates** 128).

PART III.

REACTIONS OF THE COMMONLY OCCURRING METALS, WITH THE METHODS OF THEIR SEPARATION.

GROUPING OF THE METALS.

62. The metals are divided into five groups according to their behaviour with certain substances which are termed *group reagents.* These five groups are the following :—

GROUP I.—(SILVER GROUP.)*

Group reagent, HCl.

Silver, Mercury, Lead.

The chlorides of the metals of this group are insoluble in water, and are therefore precipitated on addition of HCl.

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* A characteristic metal in each group may conveniently be used to designate that group. Thus, we may say "Silver Group" and "Copper Group," in place of Groups I. and II.

GROUP II. (COPPER GROUP.)

Group reagent, H_2S in presence of HCl.

Mercury, Lead, Bismuth, Copper, Cadmium, Arsenic, Antimony, and Tin.

The sulphides of the metals of this group are insoluble in HCl, and are therefore precipitated by H_2S in an aqueous solution containing HCl. The three last metals, arsenic, antimony, and tin, form a subgroup, as their sulphides are soluble in $(NH_4)_2S$, whilst the sulphides of the remaining metals are insoluble in that reagent.

GROUP III. (IRON GROUP.)

Group reagent, $(NH_4)_2S$, in presence of NH_4Cl and $(NH_4)HO$.

Iron, Nickel, Cobalt, Zinc, Aluminium, Manganese, and Chromium.

The sulphides and hydrated oxides of the metals of this group are insoluble in water, and are therefore precipitated on addition of the group reagent. Aluminium and chromium are precipitated as hydrated oxides, the others as sulphides.

GROUP IV. (BARIUM GROUP).

Group reagent, $(NH_4)_2CO_3$, in presence of $(NH_4)HO$ and NH_4Cl.

Barium, Strontium, Calcium.

The carbonates of the metals of this group are insoluble in water, and are precipitated on the addition of $(NH_4)_2CO_3$; as, however, they are soluble in acids, $(NH_4)HO$ must be added when the solution is acid.

GROUP V. (POTASSIUM GROUP.)

Metals unprecipitated by the above group reagents.

Magnesium, Potassium, Sodium, Ammonium.

These metals have no common precipitant, and are therefore distinguished by individual tests.

The student should at first have several metallic salts given to him, and be asked merely to determine to which of the above groups each salt belongs; he ought next to make himself familiar with the *individual tests* for each metal which follow, and then proceed to the *separations* of the different metals. It will also be well for him to attempt to frame a table of separations for each group before consulting those given in the book.

Reactions of the Metals of the Silver Group.[†]

63. Metals whose chlorides are insoluble in water, and which are precipitated on addition of the group reagent, HCl:

Silver, Mercury, Lead.

SILVER. Ag, combining weight 108.

1. HCl produces a white curdy precipitate of AgCl, insoluble in hot water and in HNO_3, but readily soluble in $(NH_4)HO$.

2. H_2S, or $(NH_4)_2S$, produces a black precipitate of Ag_2S, soluble in boiling HNO_3, with separation of sulphur.

3. NaHO produces a light brown precipitate of Ag_2O, insoluble in excess of NaHO, but soluble in $(NH_4)HO$.

*4. K_2CrO_4 produces a dark red precipitate of Ag_2CrO_4, soluble in hot HNO_3; this solution deposits on cooling an acid chromate in needle-shaped crystals.

5. KI produces a pale yellow precipitate of AgI insoluble in HNO_3.

6. Heated on charcoal with Na_2CO_3, in the reducing flame of the blow-pipe, yields bright, malleable metallic beads, soluble in HNO_3 (**56**, *a*).

64. MERCURY. Hg, c.w. 200. **Mercurous Salts.**

1. HCl produces a white precipitate of Hg_2Cl_2 (calomel), insoluble in cold HNO_3, and blackened by $(NH_4)HO$, from formation of $Hg_2Cl(NH_2)$.

[†] The best confirmatory tests are indicated in the following pages by an asterisk.

2. H_2S, or $(NH_4)_2S$, produces a black precipitate of Hg_2S, not dissolved by boiling HNO_3.

3. NaHO produces a black precipitate of Hg_2O, insoluble in excess of NaHO or $(NH_4)HO$.

*4. $SnCl_2$ produces a grey precipitate of Hg. If the fluid be poured off and the residue boiled with HCl, distinct globules are obtained.

5. KI produces a dark green precipitate of Hg_2I_2.

6. K_2CrO_4 produces an orange precipitate of mercurous chromate.

7. If a drop of neutral or only slightly acid solution of a mercurous salt be placed on a bright piece of copper, metallic mercury is deposited and the stain becomes bright on rubbing : it disappears on heating, owing to the volatility of the mercury.

8. Heated in a small tube with $NaHCO_3$, yields grey deposit of Hg, which on rubbing appears in distinct globules (**59**).

65. LEAD. Pb, C.W. 207.

1. HCl produces a white precipitate of $PbCl_2$, which is converted into a basic salt on adding ammonia, but without change of appearance. $PbCl_2$ is soluble in a small quantity of hot water, or in a large quantity of cold water.

2. H_2SO_4 produces a heavy white precipitate of $PbSO_4$, soluble in NaHO. In dilute solutions this precipitate appears only on standing; if therefore there is no immediate precipitation, the solution should be concentrated by evaporation. $PbSO_4$ is soluble in boiling HCl, and the solution on cooling deposits needle-shaped crystals of $PbCl_2$.

3. K_2CrO_4 produces a bright yellow precipitate of $PbCrO_4$, readily soluble in NaHO, but with difficulty in HNO_3.

*4. KI produces a bright yellow precipitate of PbI_2, soluble in boiling water; the solution on cooling deposits the salt in brilliant golden hexagonal scales.

5. Heated on charcoal with $NaHCO_3$, yields malleable beads, and at the same time a yellow incrustation of PbO on the charcoal (**56**, *a*).

TABLE C.

SILVER GROUP (I.).

66. Separation of Silver, Mercury, and Lead.

Add HCl and filter from the precipitated chlorides.

PRECIPITATE.	FILTRATE.
$AgCl, Hg_2Cl_2, PbCl_2$.	Groups II., III., IV. & V.

Wash precipitate twice with cold water, and add washings to filtrate, then twice with hot water, and test part of this for lead with dilute H_2SO_4. White precipitate indicates **Lead**. Boil the remaining part down to obtain the needle-shaped crystals of $PbCl_2$. If lead be found, wash the precipitate free from it with hot water, and treat the residue repeatedly with *warm* $(NH_4)HO$; filter.

RESIDUE.	FILTRATE.
Hg.	**Ag.**
If the residue is black, this indicates **Mercury.** Dissolve in HCl + HNO_3 and test with Sn Cl_2. Confirm by reduction test (**69**).	Add HNO_3. A white precipitate indicates the presence of **Silver.** Confirm with blow-pipe (**56**, *a*).

Reactions of the Metals of the Copper Group.

67. Metals whose sulphides are insoluble in HCl and are precipitated in presence of that acid by the group reagent H_2S.

Mercury, Lead, Bismuth, Copper, Cadmium, Arsenic, Antimony, and Tin.

SUB-GROUP A.—Sulphides of the above metals insoluble in $(NH_4)_2S_2$, viz., **Mercury, Lead, Copper, Bismuth,** and **Cadmium.**

MERCURY. Hg, C.W. 200. **Mercuric Salts.**

1. H_2S produces, when added by degrees, first a white precipitate, which changes to orange, then to brownish red, and finally to a black precipitate of HgS. These successive changes of colour on the addition of H_2S are exceedingly characteristic. This precipitate is insoluble in HCl and in HNO_3, even on boiling; it is soluble, however, in KHS and in *aqua regia.*

2. KHO produces a yellow precipitate of HgO, which is insoluble in excess of the precipitant, except when added to very acid solutions.

3. $(NH_4)HO$ produces in solutions of $HgCl_2$ a white precipitate of $HgCl(NH_2)$ ("white precipitate").

* 4. $SnCl_2$ produces, when added in small quantities, a white precipitate of Hg_2Cl_2, but on adding an excess of the reagent, metallic mercury precipitates as a grey powder, and may be united into a coherent globule by boiling with HCl.

5. KI produces a bright red precipitate of HgI_2 soluble in excess either of KI or of $HgCl_2$.

6. Reactions 6 and 7 for mercurous salts (**64**) are also produced with mercuric salts.

68. LEAD. Pb, c.w. 207.

1. H_2S produces a black precipitate of PbS, even in solutions of $PbCl_2$, so that a weak solution of a lead salt which has not been precipitated with HCl will be precipitated with H_2S. Hence lead occurs both in the silver and copper groups.

2. Reactions 2, 3, 4, and 5, for lead, in Group I. (**65**), are also applicable in this group.

69. BISMUTH. Bi, c.w. 210.

1. H_2S produces a black precipitate of Bi_2S_3, insoluble in KHS and KHO, but soluble in HNO_3.

2. KHO or $(NH_4)HO$ produces a white precipitate, which on boiling becomes yellow (Bi_2O_3); the precipitate is insoluble in excess of either reagent.

* 3. H_2O, when added in considerable quantity to normal salts of bismuth, produces an immediate white precipitate of a basic salt of bismuth.

Bismuth trichloride is most easily precipitated by H_2O. If another salt of this metal is being examined, it is best to precipitate the oxide first by ammonia;

dissolve it in as little HCl as possible, and evaporate it almost to dryness. On adding water to this solution, a precipitate of BiOCl at once forms, which is insoluble in tartaric acid (compare corresponding reaction with antimony, **74**, 4). Solutions of bismuth salts containing much free acid do not give this reaction with H_2O until the excess of acid has been expelled by evaporation.

4. K_2CrO_4 produces a yellow precipitate of $Bi_2(CrO_4)_3$, soluble in HNO_3, and insoluble in NaHO. (Compare reaction for lead, **65**, 3.)

5. Heated on charcoal with $NaHCO_3$ in the reducing flame of the blow-pipe, yields brittle metallic globules; also a slight yellow incrustation of oxide on the charcoal.

70. COPPER. Cu, c.w. 63·5.

1. H_2S produces a black precipitate of CuS, soluble in HNO_3, but insoluble in KHS, and only slightly soluble in $(NH_4)_2S_2$. CuS is also dissolved by KCN, but is insoluble in hot dilute H_2SO_4.

2. KHO produces a pale blue precipitate of $Cu(HO)_2$, insoluble in excess of the precipitant. If the KHO be added in excess and the mixture boiled, the precipitate becomes black and loses water.

* 3. $(NH_4)HO$ produces, when added in small quantities, a greenish blue precipitate of a basic salt, soluble in excess to a dark blue solution, which consists of a double basic salt of copper and ammonium.

4. $K_4Fe(CN)_6$ produces a brown precipitate of

$Cu_2Fe(CN)_6$, insoluble in dilute acids, but decomposed by KHO.

5. Fe precipitates copper in the metallic state, from its solutions, especially in presence of a little free acid. The iron ought to be bright and clean.

6. Zn also precipitates copper solutions. If a solution of copper containing a few drops of HCl be placed in a platinum capsule, and a fragment of zinc added, the copper will be precipitated on the platinum as a red coating.

7. Compounds of copper, when heated in the Bunsen lamp flame, impart a green colour to it, especially after addition of AgCl.

8. Mixed with $NaHCO_3 + KCN$ and heated on charcoal before the reducing blow-pipe flame, yields bright red metallic particles, soluble in HNO_3, and giving a deep blue solution on adding $(NH_4)HO$.

71. CADMIUM. Cd, c.w. 112.

1. H_2S produces a yellow precipitate of CdS, soluble in HNO_3, but insoluble in KHS, in $(NH_4)_2S$, and in KCN. CdS is dissolved by hot dilute H_2SO_4 (Compare reaction for copper, **70**, 1.)

2. KHO produces a white precipitate of $Cd(HO)_2$, insoluble in an excess of the precipitant.

3. $(NH_4)HO$ also precipitates $Cd(HO)_2$, but the precipitate is soluble in excess.

* 4. Heated on charcoal with $NaHCO_3$ in the reducing blow-pipe flame, yields no metallic bead, but a brown incrustation of CdO.

TABLE D.

72.—COPPER GROUP (II.).

Separation of Mercury, Lead, Bismuth, Copper, and Cadmium (Sub-Group A).

To the filtrate from the Silver Group add an equal bulk of HCl, boil down nearly to dryness, dilute with H_2O, and pass H_2S through the hot solution. Filter.

RESIDUE.	FILTRATE.
HgS, PbS, Bi_2S_3, CuS, CdS, SnS, SnS_2, Sb_2S_3, and **As_2S_3.**	**Groups III., IV., and V.**

Wash with hot H_2O containing H_2S, until free from Cl; digest residue* with $(NH_4)_2S_2$ for about fifteen minutes. Filter.

RESIDUE.	FILTRATE.
HgS, PbS, Bi_2S_3, CuS and **CdS.**	Sub-Group B. **(Sn, Sb, and As.)** (See Table E.)

* **In** the absence of the sub-group omit this and treat with HNO_3 as below.

Wash with hot H_2O till no longer alkaline; add a small quantity of boiling HNO_3, pouring it on several times. Filter.

RESIDUE.

HgS.

Dissolve in aqua regia, boil down to expel acid, and test with $SnCl_2$. White precipitate changing to grey indicates **Mercury.** Confirm by reduction test, or Bunsen's test for mercury (**59**).

FILTRATE.

Pb, Bi, Cu, Cd.

Add H_2SO_4, and boil down. White precipitate indicates **Lead.** Filter from $PbSO_4$, and add $(NH_4)HO$ to filtrate. Filter.

RESIDUE.

Bi.

A white precipitate. Dissolve in HCl, evaporate to a small bulk, and add to H_2O. A white precipitate indicates **Bismuth.**

FILTRATE.

Cu, Cd.

A blue solution indicates **Copper.** Adopt Method I. or II.

METHOD I.

Add to the blue solution KCN till colourless; pass a few bubbles of H_2S through the solution. Yellow precipitate indicates **Cadmium.** (Excess of H_2S must be avoided, because traces of mercury may be present.)

METHOD II.

Add HCl till acid; pass H_2S through; filter; wash rapidly and thoroughly, and treat with hot dilute H_2SO_4. Black residue indicates **Copper.** To filtrate, add H_2S. Yellow precipitate indicates **Cadmium.**

73. SUB-GROUP B.—Sulphides soluble in $(NH_4)_2S_2$, viz., those of **Tin, Antimony,** and **Arsenic.**

TIN, Sn, C.W. 118. **Stannous Salts.**

1. H_2S produces a dark brown precipitate of SnS, soluble in $(NH_4)_2S_2$ (yellow), but nearly insoluble in $(NH_4)_2S$ (colourless). From its solution in $(NH_4)_2S_2$ it is re-precipitated as SnS_2 (yellow) on adding HCl. SnS is also soluble in KHO, and, on the addition of acids, is re-precipitated as SnS (brown).

2. KHO produces a white precipitate of Sn $(HO)_2$ soluble in excess of the reagent.

3. $(NH_4)HO$ also produces a precipitate of $Sn(HO)_2$, but not soluble in excess.

* 4. $HgCl_2$ produces at first a white precipitate of Hg_2Cl_2, and on boiling with excess of the reagent, a grey precipitate of Hg.

5. $AuCl_3$ produces a purple precipitate (purple of Cassius) on addition of a little HNO_3.

6. Zn produces a precipitate of metallic tin, in shining laminæ or as a spongy mass.

7. Mixed with $NaHCO_3 + KCN$ and heated on charcoal in the reducing blow-pipe flame, yields small globules of Sn and a white incrustation of SnO_2.

Stannic Salts.—1. H_2S produces a yellow precipitate of SnS_2, soluble in $(NH_4)_2S$, in KHO, and in boiling concentrated HCl. It is with difficulty soluble in $(NH_4)HO$, and insoluble in $(NH_4)_2CO_3$.

2. KHO or $(NH_4)HO$ produces a white precipitate of $SnO(HO)_2$, soluble in an excess of the precipitant.

* 3. Zn produces the same reaction as with stannous salts. (See above.)

4. The blow-pipe reaction for Stannic is the same as for Stannous Salts. (See above.)

74. ANTIMONY. Sb, C.W. 120.

1. H_2S produces in acid solutions an orange precipitate of Sb_2S_3, soluble in alkaline sulphides, in KHO, in boiling concentrated HCl, but insoluble in $(NH_4)_2CO_3$.

2. KHO produces a precipitate of Sb_2O_3, soluble in excess of the reagent.

3. $(NH_4)HO$ produces a precipitate of Sb_2O_3, insoluble in excess of the reagent.

4. H_2O produces in solutions of $SbCl_3$ a white precipitate of SbOCl, soluble in tartaric acid : compare corresponding reaction with bismuth (**69**, 3).

5. Zn, in presence of HCl and platinum, precipitates Sb as a black powder, which adheres to the platinum This is best done either by placing a strip of Zn and Pt (in contact) in a solution of Sb made acid with HCl, or by placing a similar solution in a platinum capsule and dropping in a piece of Zn. The black stain on the platinum is not dissolved by cold HCl, but is immediately dissolved by warm HNO_3.

* 6 (Marsh's Test).—If a solution of Sb be placed in a flask along with Zn and dilute H_2SO_4, SbH_3 is given off as a gas, which is decomposed by heat, Sb being deposited. The apparatus is arranged as in the figure. *a* is the evolution flask ; *b*, a tube containing

$CaCl_2$ to absorb moisture; and *c*, a tube of hard glass attached to the drying tube, which is drawn out to a point at the extreme end, so as to form a jet. When the dilute acid and zinc have been in contact some time, and when the air has been expelled from the flask, the hydrogen evolved is lighted at the jet and a cold porcelain crucible lid is held against the flame; if a black stain be produced on it, the materials employed are not pure, and must be rejected. Having ascertained that the Zn and H_2SO_4 are pure, add now by the funnel tube a *few drops* of a solution of antimony, and observe that the flame now burns with a bluish green colour, and gives off white fumes (Sb_2O_3), and that on placing a cold porcelain lid against the flame a dull black stain of metallic antimony is deposited on it. Obtain several of these stains in order to compare them with the corresponding arsenic ones, and then heat the tube *c* with the lamp flame. Observe

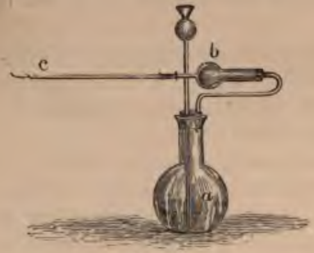

FIG. 16

the deposition of Sb *close to the flame*, and the simultaneous decrease of the green tint of the flame at the jet. Obtain several of these mirrors, observe their silvery lustre, and keep some for comparison with arsenic mirrors.

Further reactions.—(*a*) Add to the stain on porcelain a drop of NaClO : the stain will remain undissolved.

(*b*) Cut off with a file the portion of tube containing the metallic mirror, and heat it in a dry test tube. The mirror will be oxidized to Sb_2O_3, which will deposit as a sublimate on the test tube. Examine this with a lens, and ascertain that it is *amorphous*.

(*c*) Attach another tube containing an antimony mirror to an apparatus evolving dry H_2S, and warm the mirror gently (heating first the part of the mirror farthest from the evolution flask) : observe the change in colour from the formation of orange Sb_2S_3. Now detach the tube and pass through it (without heating) a current of dry HCl gas ; the Sb_2S_3 will be converted into $SbCl_3$, which is volatile, and may be collected by dipping the end of the tube under water. On adding H_2S to this liquid, orange Sb_2S_3 will be re-precipitated.

7. Allow a current of SbH_3 to pass through a solution of silver nitrate : $SbAg_3$ will be precipitated (black) and nitric acid left in solution. Filter and dissolve the residue in a hot solution of tartaric acid, add a few drops of HCl, and pass H_2S through the solution : an orange precipitate of Sb_2S_3 will be obtained.

8. Heated with $NaHCO_3$ on charcoal in the reducing blow-pipe flame, yields brittle globules of the metal

and a white incrustation of Sb_2O_3 on the charcoal. Fumes of the oxide are also given off after the metal has been removed from the flame, and they occasionally condense round the bead in a crystalline mass.

75. ARSENIC. As, c.w. 75·2.

1. H_2S produces in acid solutions a yellow precipitate of As_2S_3, soluble in alkaline sulphides, in KHO, in HNO_3, and in $(NH_4)_2CO_3$, but nearly insoluble in boiling concentrated HCl. (Compare reactions for Sb, **74,** 1.)

2. $AgNO_3$ produces in *neutral* solutions of the arsenites a pale yellow precipitate of Ag_3AsO_3. This is best obtained by adding $AgNO_3$ to an aqueous solution of As_2O_3, and then drop by drop a *very dilute solution* of (NH_4) HO prepared by adding one or two drops of ordinary (NH_4) HO to a test-tube full of H_2O. The precipitate is readily soluble in excess of $(NH_4)HO$, hence the necessity for using a very dilute solution of that reagent.

3. $CuSO_4$ added under the same conditions as the $AgNO_3$, produces a pale green precipitate of $CuHAsO_3$ (Scheele's green), soluble in $(NH_4)HO$.

4. Acetic acid, added to solutions of As_2O_3 and then KHO in slight excess, yields (after evaporation to dryness), on ignition in a small tube, oxide of cacodyl 2 $(As(CH_3)_2)O$, readily recognized by its powerful and characteristic odour. If $SnCl_2$ be added to the contents of the tube after ignition, the equally characteristic smell of cacodyl chloride, As $(CH_3)_2Cl$, is ob-

served. These experiments (and also Marsh's test (5)) must be done with an *exceedingly small* quantity of substance, owing to the poisonous properties of the products.

*5. Proceed exactly as in Marsh's test for Sb (**74**, 6), substituting a solution of As for one of Sb, and observe the bluish flame with which the mixture of H and AsH_3 burns, and also the production of white fumes of As_2O_3. Obtain, as in the case of Sb, stains on porcelain lids, and mirrors by heating the hard glass tube. Compare these with the antimony stains and mirrors, and observe that the latter in the case of As are deposited at a greater distance from the heated part of the tube, owing to the greater volatility of As. Observe also the distinction in colour of the stains: dark brown or almost black in the case of Sb, and, when seen in thin films, pale brown and lustrous in the case of As.

Further reactions.—(*a*) Add to one of the stains on porcelain a drop of NaClO: it will be rapidly dissolved.

(*b*) Cut off the portion of tube containing a metallic mirror, and heat it in a dry test tube. The mirror of As will be oxidized to As_2O_3, which will be deposited in crystals on the cool part of the tube. Examine these with a lens, and observe the octahedral form of the crystals. Take out the piece of tubing which contained the mirror, and dissolve the crystals left in the test-tube in warm H_2O; add to this solution $AgNO_3$ and very dilute $(NH_4)HO$, and observe the yellow precipitate of Ag_3AsO_3. Or, to the aqueous solution of

As_2O_3 add a drop of HCl, and pass H_2S through the solution, and observe the yellow precipitate of As_2S_3.

(*c*) Attach another tube containing an arsenic mirror to an apparatus evolving dry H_2S, and warm the mirror gently : it will be converted into yellow As_2S_3. Now pass dry HCl through the tube (without warming), and observe that the sulphide remains unaltered. (Compare corresponding Sb reactions, **74**, 6, *c*).

(*d*) Allow a current of AsH_3 to pass through a solution of silver nitrate : a black precipitate of Ag will be produced, and As_2O_3 will be found in solution along with HNO_3 liberated from the $AgNO_3$. Filter from the Ag, and very cautiously neutralize the free acid with highly diluted $(NH_4)HO$, when a yellow precipitate of Ag_3AsO_3 will be formed. (Compare corresponding Sb reaction, **74**, 7.)

6. Compounds of arsenic, when treated with Zn and strong solution of KHO, are converted into AsH_3 by the action of the nascent hydrogen. If this reaction be performed in a test tube, and the gas escaping be held near a piece of paper moistened with $AgNO_3$, a bluish black coloration is produced by the formation of $AsAg_3$. (Sb compounds give no similar reaction.)

7 (Reinsch's Test).—Add to the solution of arsenic, HCl and a few strips of bright copper wire or foil : As is deposited on the copper, which may be removed from the solution, dried by filter paper, and heated in a dry test-tube to obtain the octahedral crystals of As_2O_3.

8. Dry reactions.

Place the dry arsenic compound at *a* (Fig. 17), in a drawn-out hard glass tube. Then place above it at *b* a small rod of well-ignited charcoal, and heat the portion containing the charcoal until it is red hot. This will cause the glass to soften, and the tube will bend so as to bring the portion *a* into the flame. The arsenic compound will volatilize and be decomposed by the red-hot charcoal, and a metallic mirror will form at *c*.*

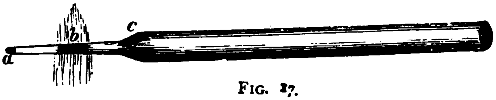

FIG. 17.

9. Place the dry arsenic compound in a bulb tube as at *a* (Fig. 18), along with a mixture of equal parts of *dry* Na_2CO_3 and KCN, and heat the bulb. A mirror of As will form at *b*, which may be further tested by the reactions mentioned for the mirror obtained in Marsh's test (**75**, 5, *b*). If any moisture be deposited on first heating the tube, remove it by inserting a small coil of filter paper.

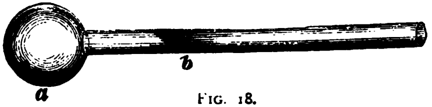

FIG. 18.

10. The above reaction is more delicate when the mixture is heated in a current of dry CO_2. For this

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* Non-volatile compounds of As must be mixed with dry charcoal powder, and heated in a similar tube, having in addition a small bulb at the lower end to contain the mixture

purpose, the arsenic compound is pounded in a mortar with a perfectly dry mixture of three parts Na_2CO_3 with one part of KCN, and placed at *a* in the tube *ab* (Fig. 19), through which a slow current of *dry* CO_2 is led, and the whole tube *gently* heated until every trace of moisture is expelled. When this is the case the tube is more strongly heated at *a*, and the mirror is obtained at *b*; traces of arsenic escape condensation, and therefore a slight garlic odour is observed at the extremity of the tube. Antimony compounds treated in this way yield no metallic mirror.

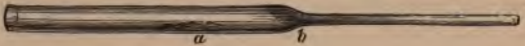

FIG. 19.

11. Arsenic compounds, when mixed with Na_2CO_3 and heated on charcoal by the blow-pipe flame, are reduced to metallic arsenic, which at once volatilizes, and may be recognized by the characteristic odour resembling garlic.

TABLE E.

GROUP II.

76. Separation of Arsenic, Antimony, and Tin (Sub-Group B).

Solution in $(NH_4)_2S_2$ contains sulphides of As, Sb, and Sn. Add HCl until acid: the metals are re-precipitated as sulphides. Filter, wash precipitate with hot water till free from HCl; digest precipitate with one or two pieces of solid $(NH_4)_2CO_3$ and H_2O. **Filter.**

RESIDUE.	FILTRATE.
SnS_2, Sb_2S_3.	**As.**
Wash and dissolve in strong boiling HCl. dilute with water, filter, and divide the filtrate into two parts. In one, place a piece of platinum foil and a fragment of zinc touching it. **Sb** forms a black stain on the platinum. Dissolve by warming with a few drops of HNO_3, dilute with water and add H_2S. An orange precipitate indicates **Antimony.** Boil the other portion of the filtrate *for at least five minutes* with some metallic copper. Pour off the liquid and add $HgCl_2$. White or grey precipitate indicates **Tin.**	Add HCl until acid; wash precipitated sulphide, and dissolve in HCl and a little $KClO_3$, boil down to a small bulk, and apply Marsh's test (**75**, 5). Metallic mirror, yielding octahedral crystals on heating, indicates **Arsenic.** Dissolve in H_2O, and confirm by adding $AgNO_3$ and dilute $(NH_4)HO$, to obtain yellow precipitate of Ag_3AsO_3 (**75**, 5 *b*).

Reactions of the Metals of the Iron Group.

77. Metals whose sulphides and hydrated oxides are insoluble in water, and are precipitated on addition of the group reagent $(NH_4)_2S$ in presence of $(NH_4)HO$ and NH_4Cl.

Iron, Nickel, Cobalt, Zinc, Aluminium, Manganese, and Chromium.

IRON. Fe, C.W. 56. **Ferrous Salts.**

1. $(NH_4)_2S$ produces a black precipitate of FeS, insoluble in alkalies, but soluble in HCl. In dilute solutions of ferrous salts $(NH_4)_2S$ produces at first a green colour; on standing, however, FeS separates as a black precipitate.

2. KHO or $(NH_4)HO$ produces a white precipitate of ferrous hydrate $Fe(HO)_2$, which rapidly acquires a dirty green, and ultimately a reddish brown colour,

owing to absorption of oxygen and conversion into ferric hydrate, $Fe_2(HO)_6$.

3. Carbonates of the alkalies precipitate $FeCO_3$ (white), which rapidly darkens in colour owing to absorption of oxygen.

4. $K_4Fe(CN)_6$ produces a white precipitate of $K_2Fe_2(CN)_6$, which rapidly becomes blue by oxidation to $Fe_5(CN)_{12}$ (Prussian blue). It is insoluble in acids, but is decomposed by alkalies.

*5. $K_3Fe(CN)_6$ produces a blue precipitate of $Fe_3 Fe_2(CN)_{12}$ (Turnbull's blue), also insoluble in acids, but decomposed by alkalies.

6. KCNS produces no coloration.

7. Ba CO_3 produces no precipitate in cold solutions of ferrous salts.

8. Fused with borax in the oxidizing flame, yellowish red beads are produced; in the reducing flame the beads become green. (See also **59.**)

Ferric Salts.—1. H_2S in acid solutions produces a precipitate of sulphur, and the salt is reduced to protosalt, thus: $Fe_2Cl_6 + H_2S = 2\,FeCl_2 + 2\,HCl + S$.

2. $(NH_4)_2S$ produces a black precipitate of FeS mixed with sulphur, insoluble in excess of the reagent and in alkalies, but soluble in HCl and in HNO_3. In dilute solutions of iron only a greenish coloration is produced.

3. KHO or $(NH_4)HO$ produces a brownish red precipitate of $Fe_2(HO)_6$, insoluble in excess of either reagent.

*4. $K_4Fe(CN)_6$ produces a precipitate of $Fe_5(CN)_{12}$ (Prussian blue), insoluble in HCl, soluble in $C_2H_2O_4$, and decomposed by KHO or NaHO with separation of $Fe_2(HO)_6$.

5. $K_3Fe(CN)_6$ changes the colour of the solution to reddish brown, but does not produce a precipitate.

6. KCNS produces even in dilute solutions a blood-red coloration, due to the formation of a soluble iron sulphocyanide. HCl does not destroy the coloration, but it is destroyed by $C_2H_3O_2Na$, $HgCl_2$, H_3PO_4, and by $C_4H_6O_6$.

7. $BaCO_3$ precipitates ferric solutions completely as $Fe_2(HO)_6$ mixed with basic salt.

8. The blow-pipe reactions are the same as for ferrous compounds.

78. NICKEL. Ni, c.w. 58·7.

1. $(NH_4)_2S$ produces a black precipitate of NiS, slightly soluble in excess, forming a brown solution from which NiS is precipitated on boiling. The precipitate is very difficultly soluble in HCl, but dissolves in HNO_3 and in aqua regia.

2. NaHO or KHO produces a light green precipitate of $Ni(HO)_2$ insoluble in excess of the reagent, and unalterable in air.

3. $(NH_4)HO$ produces also a precipitate of $Ni(HO)_2$, readily soluble in excess, yielding a blue fluid, which is re-precipitated by KHO or NaHO. Acid solutions, or those containing salts of ammonia, yield no precipitate with $(NH_4)HO$.

*4. KCN produces a yellowish green precipitate of $Ni(CN)_2$, soluble in excess to a brownish yellow solution of 2 KCN, $Ni(CN)_2$. This solution is re-precipitated by addition of dilute HCl or H_2SO_4, and, if boiled with a strong solution of NaClO, yields a black precipitate of $Ni_2(HO)_6$.

5. KNO_2, in presence of $C_2H_4O_2$, produces no precipitate.

6. Fused with borax in the oxidizing blow-pipe flame, yields reddish yellow beads when hot, which become paler on cooling. In the reducing flame the bead becomes grey by the presence of metallic nickel. (See also **59**.)

79. COBALT. Co, c.w. 58·7.

1. $(NH_4)_2S$ produces a black precipitate of CoS, insoluble in excess of the reagent and in HCl, but soluble in aqua regia.

2. KHO or NaHO precipitates blue basic salts, which turn green on exposure to air by oxidation. On heating the precipitate is converted into red hydrate $Co(HO)_2$, which is soluble in $(NH_4)_2CO_3$ to a reddish violet solution.

3. $(NH_4)HO$ produces the same precipitate as KHO, soluble in excess, yielding a reddish brown fluid, which is re-precipitated by KHO or NaHO. Acid solutions, or those containing salts of ammonia, are not precipitated.

4. KCN produces a light brown precipitate of $Co(CN)_2$, soluble in excess of the reagent by formation of $2KCN, Co(CN)_2$. This solution is re-precipitated by the addition of HCl or H_2SO_4. (If the cobalt solution to which KCN is added be acid, a precipitate is produced, soluble in excess of the reagent. When this solution is boiled, potassium cobalti-cyanide $K_3Co(CN)_6$ is formed which is not re-precipitated by HCl or H_2SO_4, nor by NaClO.)

5. KNO_2, added to cobalt solutions with addition of acetic acid, produces on standing in a warm place a yellow crystalline precipitate (double nitrite of potassium and cobalt).

* 6. Fused with borax in either blow-pipe flame, yields deep blue beads, which are almost black if the quantity of Co be large. (See also **59.**)

80. ZINC. Zn, c.w. 65·2.

1. $(NH_4)_2S$ produces a white precipitate of ZnS, insoluble in excess of the reagent and in KHO, but soluble in the mineral acids.

2. KHO or NaHO produces a white precipitate of $Zn(HO)_2$, soluble in excess of either reagent and in $(NH_4)HO$. This solution is re-precipitated by diluting and boiling, but is not precipitated by addition of NH_4Cl.

3. Na_2CO_3 produces a white precipitate of basic carbonate, insoluble in excess of the reagent.

4. $(NH_4)_2CO_3$ also precipitates the basic carbonate, but it is soluble in excess of the reagent.

5. Heated on charcoal with Na_2CO_3 in the reducing blow-pipe flame, a yellow incrustation of ZnO is obtained, which becomes white when cold.

* 6. Heated on charcoal by the blow-pipe flame after moistening with $CoCl_2$ solution, an infusible green mass is obtained. (See also **59.**)

81. ALUMINIUM. Al, c.w. 27·3.

1. $(NH_4)_2S$ produces a white flocculent precipitate of $Al_2(HO)_6$.

2. KHO or NaHO produces also a precipitate of $Al_2(HO)_6$, soluble in acids, even in hot acetic acid and in excess of the reagent. This solution is not precipitated by H_2S, but is re-precipitated by NH_4Cl, or by adding $(NH_4)HO$ after acidifying with HCl.

3. $(NH_4)HO$ also precipitates $Al_2(HO)_6$, soluble in a very large excess of the reagent, more difficultly soluble in presence of salts of ammonia.

4. $BaCO_3$ produces a precipitate of $Al_2(HO)_6$ mixed with basic salt.

5. Na_2HPO_4 precipitates aluminium phosphate, insoluble in $(NH_4)HO$ and in NH_4Cl, but soluble in KHO or NaHO, and in acids. It does not, however, dissolve in hot acetic acid like aluminium hydrate.

* 6. Heated on charcoal in the blow-pipe flame, then moistened with $CoCl_2$, and re-heated, an infusible blue mass is obtained.

82. MANGANESE. Mn, c.w. 55.

1. $(NH_4)_2S$ produces a flesh-coloured precipitate of MnS, soluble in acids, even in acetic acid.

2. KHO or NaHO produces a dirty-white precipitate of $Mn(HO)_2$, insoluble in excess of the reagent; the precipitate rapidly darkens in colour by absorption of oxygen. The freshly-precipitated hydrate is dissolved by NH_4Cl, but the higher oxide is insoluble.

3. $(NH_4)HO$ produces the same precipitate of $Mn(HO)_2$, insoluble in excess of the reagent; but it gives no precipitate if the manganese solution contain NH_4Cl. Such a solution on standing precipitates the dark brown hydrate.

4. Na_2CO_3 produces a white precipitate of $MnCO_3$, which darkens in colour by absorption of oxygen.

5. If any manganese solution (free from chlorine) be treated with PbO_2 and then boiled with HNO_3, it is converted into permanganate, which is recognized by its pink colour as soon as the mixture has settled.

* 6. If any manganese compound be fused on platinum foil with Na_2CO_3 and a trace of KNO_3, it is converted into Na_2MnO_4, recognized by its bright green colour.

7. Fused with borax in the oxidizing flame, an amethyst-coloured bead is obtained, which becomes colourless in the reducing flame.

83. CHROMIUM. Cr, c.w. 52·1.

1. $(NH_4)_2S$ produces a bluish green precipitate of $Cr_2(HO)_6$, insoluble in excess of the reagent.

2. $(NH_4)HO$ also precipitates the hydrate, soluble to some extent in excess, yielding a pink fluid, but on heating the precipitation is complete.

3. KHO or NaHO precipitates also $Cr_2(HO)_6$, soluble however in excess, yielding a green or bluish violet solution. On continued boiling or addition of NH_4Cl and heating, the hydrate is re-precipitated.

4. $BaCO_3$ produces a precipitate of $Cr_2(HO)_6$ along with basic salt; the precipitation is not complete till the mixture has stood some time.

5. Fused with Na_2CO_3 and KNO_3 on platinum foil, yellow Na_2CrO_4 is obtained.

* 6. Fused with borax in either flame (but best in the reducing flame), green beads are obtained.

84. IRON GROUP*(III.).—**Separation of Iron, Nickel,**

To the filtrate from the sulphides of the Cu and As groups add $(NH_4)HO$ and shake for some time. Filter (preferably by the Bunsen pump). Wash filtrate is often brown in presence of Ni. Treat

<table>
<tr><td>RESIDUE.</td><td colspan="3"></td></tr>
<tr><td rowspan="6">NiS and CoS.
Test for Co by borax bead. Dissolve the black residue in HCl and $KClO_3$. Boil down just to dryness, dilute with H_2O, add KCN in excess, then a drop of acetic acid, boil for a few minutes, add $NaClO$† in excess, and boil again. A black precipitate indicates Nickel. The filtrate from this precipitate may be tested for Co by evaporating to dryness, and fusing in a borax bead. Blue colour indicates Cobalt.</td><td colspan="3"></td></tr>
<tr><td colspan="3"><i>METHOD I.</i> Cr IS ABSENT.
Boil down with a little $KClO_3$ till it smells of Cl. Add pure $NaHO$ till strongly alkaline. Filter.</td></tr>
<tr><td colspan="2">RESIDUE.</td><td>FILTRATE.</td></tr>
<tr><td colspan="2">$Fe_2(HO)_6$, $Mn(HO)_2$.
Wash with hot H_2O, dissolve in HCl, add $(NH_4)HO$, and filter.</td><td rowspan="3">Al, Zn.
Divide into two parts.
1. Add H_2S or $(NH_4)_2S$. A white precipitate indicates Zinc. Confirm by flame-reaction.
2. Add HCl till acid, then $(NH_4)HO$ till alkaline. A white precipitate indicates Aluminium. Confirm by flame reaction.</td></tr>
<tr><td>RESIDUE.</td><td>FILTRATE.</td></tr>
<tr><td>$Fe_2(HO)_6$.
Dissolve in HCl. Test with $K_4Fe(CN)_6$. Blue precipitate indicates Iron. To ascertain whether the iron is present as ferrous or ferric salt, the <i>original solution</i> must be tested with $K_4Fe(CN)_6$, and $K_3Fe(CN)_6$.</td><td>Mn.
Boil down and ignite, to expel salts of ammonium. Fuse with $NaHO$ and KNO_3. A green residue indicates Manganese. Traces of Ni and Co are found along with the Mn.</td></tr>
</table>

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* For the separation of the metals of this group in presence of Phosphoric Acid, &c., see Table L in the Appendix.

† $NaHO$ and Bromine water may be used instead of $NaClO$.

F.

Cobalt, Aluminium, Zinc, Manganese, and Chromium.

(till alkaline) + (NH_4Cl + $(NH_4)_2S$. Warm the mixture gently in a small flask well with H_2O, containing $(NH_4)_2S$, and finally once with H_2O alone. The the precipitate with cold dilute HCl, and filter.

FILTRATE.

Cr, Al, Fe, Zn, and **Mn.**

(Green or violet if Cr be present. Boil down a portion and test for Cr by borax bead. Adopt Method I. if absent, Method II. if present.)

METHOD II. Cr IS PRESENT.

Boil down with a little $KClO_3$ till it smells of Cl. Add Na_2CO_3 or NaHO till just neutral or slightly acid; allow to become *perfectly* cold. Add excess of $BaCO_3$, place in a flask, cork up and shake well, allow to stand till clear. Filter.

RESIDUE.

$Fe_2(HO)_6$, $Cr_2(HO)_6$, $Al_2(HO)_6$ (also excess of $BaCO_3$).

Wash well, boil with pure NaHO, and filter; add HCl to the filtrate, and then $(NH_4)HO$ till alkaline. A white precipitate indicates **Aluminium.** Confirm by flame-reaction. Fuse the residue insoluble in NaHO with a mixture of Na_2CO_3 and KNO_3, extract with water, and filter.

RESIDUE.

$Fe_2(HO)_6$.

Dissolve in H Cl, and test with $K_4Fe(CN)_6$. A blue precipitate indicates **Iron.**

FILTRATE.

Cr.

Yellow in colour. Acidify with acetic acid; add lead acetate. A bright yellow precipitate indicates **Chromium.**

FILTRATE.

Zn, Mn.

Precipitate the Ba present with H_2SO_4 in the hot solution. Boil well, and filter; add NaHO. Precipitate indicates **Manganese.** Confirm by fusing with Na_2CO[illegible] and KNO_3 [illegible] platinum foil [illegible] the filtrate f[illegible] the $Mn(HO)_2$ add $(NH_4)_2S$. A white precipitate indicates **Zinc.** Confirm by flame-reaction.

*ANOTHER METHOD.**

Boil filtrate from Cu group to expel H_2S, then boil with $KClO_3$, add NH_4Cl, and then $(NH_4)HO$ till alkaline. Filter.

RESIDUE.

Fe, Al, Cr.

Test washed ppt. for Cr by borax bead, and for Fe with $K_4Fe(CN)_6$. Treat residue with warm NaHO, and test for Al in filtrate as in Method I. 2.

FILTRATE.

Co, Ni, Mn, Zn.

Add $(NH_4)_2S$ and filter. Separate Co and Ni with HCl as above. Boil filtrate from NiS and CoS, add NaHO till alkaline, and separate Mn and Zn as in Method II.

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* When this method is used the filtrate cannot be afterwards tested for potassium.

Reactions of the Metals of the Barium Group.

85. Metals whose carbonates are insoluble in water, and whose solutions are precipitated on the addition of $(NH_4)_2CO_3$: as, however, the carbonates are soluble in acids, the solution, if acid, must be neutralized by addition of $(NH_4)HO$.

Barium, Strontium, Calcium.

BARIUM. Ba, c.w. 137.

1. $(NH_4)_2CO_3$ produces a white precipitate of $BaCO_3$, soluble in acids, and to a slight extent in NH_4Cl.

2. K_2CO_3 or Na_2CO_3 produces also a precipitate of $BaCO_3$, insoluble in excess of either reagent.

3. H_2SO_4 or any soluble sulphate produces, even in dilute solutions, a heavy white precipitate of $BaSO_4$, insoluble in acids, alkalies, or salts of ammonium.

4. $CaSO_4$ or $SrSO_4$ produces an immediate white precipitate of $BaSO_4$.

5. H_2SiF_6 produces a white precipitate of $BaSiF_6$.

6. $C_2(NH_4)_2O_4$ produces a white precipitate of C_2BaO_4, soluble in HCl and in HNO_3.

* 7. K_2CrO_4 produces a yellow precipitate of $BaCrO_4$, insoluble in $C_2H_4O_2$, but soluble in HCl and HNO_3.

8. Heated in the lamp flame a green coloration is produced, especially on moistening the salt with HCl.

86. STRONTIUM. Sr, c.w. 87·5.

1. $(NH_4)_2CO_3$ or K_2CO_3 precipitates white $SrCO_3$, soluble in acids, but less soluble in NH_4Cl than $BaCO_3$.

2. H_2SO_4 produces a white precipitate of $SrSO_4$, which is much less insoluble in H_2O than $BaSO_4$, and it therefore precipitates from dilute solutions only on standing or warming. $SrSO_4$ is slightly soluble in HCl.

* 3. $CaSO_4$ produces, after standing some time, a white precipitate of $SrSO_4$.

4. H_2SiF_6 does not precipitate strontium solutions.

5. $C_2(NH_4)_2O_4$ produces a white precipitate of C_2SrO_4, soluble in HCl and in HNO_3, also to a slight extent in NH_4Cl, but very sparingly in $C_2H_4O_2$.

6. K_2CrO_4 produces, only in concentrated solutions, a yellow precipitate of $SrCrO_4$, soluble in $C_2H_4O_2$.

7. Heated in the lamp flame a crimson coloration is produced, especially on moistening the salt with HCl.

87. CALCIUM. Ca, c.w. 40.

1. $(NH_4)_2CO_3$ or K_2CO_3 produces a white precipitate of $CaCO_3$, which becomes crystalline on heating.

2. H_2SO_4 precipitates from strong solutions of calcium salts $CaSO_4$, as a white precipitate, which dissolves in a large excess of water, and also in acids.

3. $CaSO_4$ produces no precipitate.

4. H_2SiF_6 produces no precipitate.

* 5. $C_2(NH_4)_2O_4$ produces, even in dilute solutions, a white precipitate of C_2CaO_4, soluble in HCl or HNO_3, but insoluble in $C_2H_2O_4$ or in $C_2H_4O_2$.

6. Heated in the lamp flame, a dull red coloration is produced, especially on moistening the salt with HCl. This reaction is imperceptible in presence of Ba or Sr salts.

TABLE

88.—CALCIUM GROUP (IV.).

Separation of Barium,

Heat filtrate from iron group, add to the hot solution NH_4Cl and
and add to a portion $CaSO_4$ solution. An immediate precipitate indicates
dilute Barium solution. (Test another portion with $SrSO_4$ for Ba) To
and Sr. Filter. Neutralize filtrate with $(NH_4)HO$, and add $C_2(NH_4)_2O_4$.

TO TEST FOR

I. Ba Present. Ca Absent.

Dissolve the carbonate in HCl, and evaporate to dryness. Treat the residue with strong alcohol. Filter.

Residue.	Filtrate.
$BaCl_2$.	**Sr.**
Confirm by flame test. Green coloration indicates **Barium.**	Confirm by lighting the alcoholic solution. Crimson coloration indicates **Strontium.**

II. Ba Absent.

Dissolve as before, and
Filter, and

Residue.

$SrSO_4$.

If small, burn the filter in the reducing gas flame to convert $SrSO_4$ into SrS: moisten with HCl, and test in the lamp-flame. Crimson coloration indicates **Strontium.**

ANOTHER

Dissolve in HNO_3,
ness. Treat with strong

Residue.

$Sr(NO_3)_2$.

Confirm as above.

G.

Strontium, and Calcium.

$(NH_4)_2CO_3$, and filter. Wash precipitate with hot H_2O, dissolve in HCl, **Barium**; a precipitate after some time indicates **Strontium,** or a another portion of the solution in HCl add H_2SO_4, and boil to remove Ba An immediate precipitate indicates **Calcium.**

STRONTIUM.

Ca PRESENT.

precipitate with H_2SO_4. wash well.

FILTRATE.

Ca.

Neutralize the solution with $(NH_4)HO$, and test with $C_2(NH_4)_2O_4$. White precipitate indicates **Calcium.**

METHOD.

and evaporate to dry- alcohol. Filter.

FILTRATE.

Ca.

Confirm as above.

III. Ba AND Ca PRESENT.

Add H_2SO_4 to the HCl solution (diluted to prevent precipitation of Calcium), and filter.

RESIDUE.	FILTRATE.
Ba SO$_4$, Sr SO$_4$.	**Ca.**
Boil in a beaker with a little water, together with a mixture of three parts K_2SO_4 and one part K_2CO_3. Filter, and treat residue with HNO_3. The $SrSO_4$ is dissolved, and the $BaSO_4$ left undissolved. (Traces of Ca may be found with the Sr.)	Neutralize the solution with $(NH_4)HO$, and test with $C_2(NH_4)_2O_4$ White precipitate indicates **Calcium.**

ANOTHER METHOD.

Dissolve the carbonates in $C_2H_4O_2$, and precipitate the Ba with K_2CrO_4. Filter. Precipitate the Sr and Ca by $(NH_4)_2CO_3$, and proceed as in Method II. (Ba Absent, Ca Present).

Reactions of the Metals of the Potassium Group.

89. Metals whose solutions are unprecipitated by the preceding group reagents, but which have no common precipitant, and are therefore recognized by individual tests.

Magnesium, Potassium, Ammonium, Sodium.

MAGNESIUM. Mg, c.w. 24.

1. Na_2HPO_4 produces, in presence of $(NH_4)HO$ and NH_4Cl, a crystalline white precipitate of $MgNH_4PO_4$. From dilute solutions the precipitation is slow, but may be hastened by stirring with a glass rod. The precipitate is soluble in dilute mineral acids and in $C_2H_4O_2$, but is almost insoluble in dilute solution of $(NH_4)HO$.

2. $(NH_4)HO$ in neutral solutions produces a partial precipitation of the hydrate $Mg(HO)_2$, but gives no precipitate in presence of NH_4Cl, in which the hydrate is readily soluble.

3. H_2SO_4, H_2SiF_6, and $C_2(NH_4)_2O_4$ yield no precipitates.

*4. Heated on charcoal in the blow-pipe flame, then moistened with $CoCl_2$ and re-heated, a pink mass is obtained.

90. POTASSIUM. K, c.w. 39·1.

1. $PtCl_4$ produces a crystalline yellow precipitate of $2\,KCl + PtCl_4$, except in dilute solutions, which are not precipitated. The precipitation is promoted by stirring, or by addition of alcohol.

2 Tartaric acid precipitates white crystalline hydro-

gen potassium tartrate from strong and neutral solutions. The precipitation is promoted by stirring.

3. H_2SiF_6 produces a white gelatinous precipitate of K_2SiF_6.

* 4. Heated on platinum wire in the non-luminous flame a violet coloration is produced, which when viewed through a piece of blue glass appears reddish violet.

91. AMMONIUM. NH_4, c.w. 18.

1. $PtCl_4$ produces a crystalline yellow precipitate of $2\,NH_4Cl + PtCl_4$, except in dilute solutions, which are not precipitated. The precipitate is insoluble in alcohol and ether, and when ignited leaves a residue of spongy platinum.

2. Tartaric acid produces, in strong solutions, a white precipitate of hydrogen ammonium tartrate, similar in appearance to the corresponding potassium salt.

3. Nessler's solution, added to ammonia or its salts, produces a yellow coloration, or, if the ammonia compound be present in large quantity, a brown precipitate.

* 4. NaHo or KHO solution when warmed with ammonia salts decomposes them, and NH_3 is evolved, which is recognized by its pungent odour, by its turning red litmus paper blue, and by its forming white fumes with a strong solution of any volatile acid, *e.g.* HCl.

5. Heated on platinum foil, all compounds of ammonia volatilize completely.

92. SODIUM. Na, c.w. 23.

The soda salts are almost without exception soluble in water, so that the flame test alone serves to distinguish the salts of this metal.

* 1. Heated on platinum foil or wire in the non-luminous lamp flame, an intense yellow colour is produced, which, however, is not seen when viewed through blue glass. It is thus possible to distinguish potassium salts when mixed with sodium salts.

TABLE H.

GROUP V.

93 Separation of Magnesium, Potassium, Sodium, and Ammonium.

The filtrate from the Barium Group is concentrated by evaporation, and a portion ignited on platinum foil. If no residue is left on ignition, Mg, K, and Na are absent.

Detection of NH_4.	**Detection of Mg.**	**Detection of K and Na.**	
		(1.) **Mg. being absent.**	(2.) **Mg. being present.**
The original substance or solution is heated with $NaHO$ in a test tube. Presence of **Ammonium** shown by smell, by the white fumes with HCl, and by its action on red litmus paper.	To a portion of the concentrated cold solution add $(NH_4)HO$ and Na_2HPO_4. White crystalline precipitate denotes **Magnesium.**	Evaporate another portion of the solution to dryness, ignite residue, dissolve in a small quantity of water, filter if required, and add to the clear liquid $PtCl_4$, evaporate nearly to dryness, and add alcohol. Yellow precipitate indicates **Potassium.**	Evaporate the solution to dryness, ignite residue, dissolve in water, and add baryta water until the solution has an alkaline reaction; boil; filter. To filtrate, add $(NH_4)_2CO_3$, heat, filter, evaporate to dryness, and test the residue for **K** and **Na.** As Sub. (I.)
To detect Na. Evaporate alcoholic solution (which must have a yellow colour, showing that excess of $PtCl_4$ has been added) nearly to dryness, add a grain or two of sugar, and ignite residue. Exhaust with water, filter, evaporate to dryness; and if a residue be left, test it by flame reaction for **Na.** Yellow coloration indicates **Sodium.**			

PART IV.

REACTIONS OF THE ACIDS.

94. *Grouping of the Acids.*

The acids do not admit of being grouped with the same precision as the bases, but they can be approximately classified by means of certain group reagents. They are divided into two great classes, *Inorganic* and *Organic Acids.* These are readily distinguished by the action of heat.

Salts of *Inorganic Acids* when heated to redness are not charred; salts of *Organic Acids* are at once charred, owing to decomposition and consequent separation of carbon.*

95. Grouping of the Inorganic Acids.

GROUP I. (SULPHURIC ACID GROUP).

Group reagent, $BaCl_2$ in presence of HCl.

Sulphuric Acid, Hydrofluo-silicic Acid.

The acids of this group are precipitated by $BaCl_2$, and the precipitate is not dissolved on addition of HCl.

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* With the exception of acetic and formic acids. (See **128** and **129**

GROUP II. (PHOSPHORIC ACID GROUP).

Group reagent, $BaCl_2$.

Phosphoric, Boric, Oxalic, Hydrofluoric, Carbonic, Silicic, Sulphurous, Hyposulphurous, Arsenious, Arsenic, Iodic, and Chromic Acids.

The acids of this group are precipitated in *neutral solutions* by $BaCl_2$.

GROUP III. (HYDROCHLORIC ACID GROUP)

Group reagent, $AgNO_3$.

Hydrochloric, Hydrobromic, Hydriodic, Hydrocyanic, Hypochlorous, Nitrous, and Hydrosulphuric Acids.

The acids of this group are precipitated by $AgNO_3$, and *not* by $BaCl_2$.

GROUP IV. (NITRIC ACID GROUP).

Nitric, Chloric, and Perchloric Acids.

These acids are not precipitated by any reagent, as all their salts are soluble in water.

Reactions of the Inorganic Acids belonging to Group I.

96. Acids precipitated by $BaCl_2$ in presence of HCl.

Sulphuric Acid, Hydrofluo-silicic Acid.

SULPHURIC ACID. H_2SO_4, c.w. 98.

1. $BaCl_2$ produces a white precipitate of $BaSO_4$, insoluble in HCl or HNO_3. In very dilute solutions the precipitation is not immediate, but on standing the solution becomes clouded, and ultimately the precipitate subsides.

*2. $Pb(NO_3)_2$ produces a heavy white precipitate of $PbSO_4$, soluble in NaHO, and in boiling HCl (on allowing this solution to cool, $PbCl_2$ crystallizes out).

3. Fused on charcoal with Na_2CO_3 in the reducing flame of the blow-pipe, a sulphide is produced. If the fused mass be moistened with HCl, the odour of H_2S is at once perceptible; or if it be placed on a bright piece of silver and moistened with water, a black stain of Ag_2S is produced. As the latter reaction is very delicate, care must be taken to use Na_2CO_3 perfectly free from Na_2SO_4, and it must be borne in mind that other sulphur acids give the same reaction when heated with Na_2CO_3.

97. HYDROFLUO-SILICIC ACID. H_2SiF_6, c.w. 144.

1. $BaCl_2$ produces a crystalline precipitate of $BaSiF_6$, insoluble in HCl.

2. KCl produces a gelatinous precipitate of K_2SiF_6.

3. Heated with sulphuric acid in a plantium or leaden crucible covered with a watch-glass, the latter is etched owing to the disengagement of HF.

Reactions of the Acids belonging to Group II.

98. Acids precipitated by $BaCl_2$ in neutral solutions.

Phosphoric, Boric, Oxalic, Hydrofluoric, Carbonic, Silicic, Sulphurous, Hyposulphurous, Arsenious, Arsenic, Iodic, and Chromic Acids.

PHOSPHORIC ACID. H_3PO_4, C.W. 98. (Ortho-phosphoric Acid.)

1. $BaCl_2$ produces a white precipitate of $BaHPO_4$, readily soluble in HNO_3 or HCl, but with difficulty in NH_4Cl.

2. $MgSO_4$ along with $(NH_4)HO$ and NH_4Cl, produces a white crystalline precipitate of $Mg(NH_4)PO_4 + 6H_2O$, insoluble in $(NH_4)HO$, but soluble in HCl, HNO_3, and acetic acid. In dilute solutions the precipitation does not take place till after the lapse of some time, but is promoted by stirring and gentle warming.

3. $AgNO_3$ produces a yellow precipitate of Ag_3PO_4, soluble in HNO_3, and also in $(NH_4)HO$.

4. Lead acetate produces a white precipitate of $Pb_3(PO_4)_2$, soluble in HNO_3, but almost insoluble in acetic acid.

5. Fe_2Cl_6, in presence of excess of sodium acetate,

produces a yellowish precipitate of $FePO_4$, soluble in HCl, and in excess of Fe_2Cl_6, which ought therefore to be added drop by drop.*

* 6. Ammonium molybdate produces in solutions made acid with HNO_3 a yellow coloration and ultimately a precipitate. The reaction is hastened by warming the mixture.

The following acids of phosphorus are distinguished from each other and from ortho-phosphoric acid by the following reactions:—

Pyrophosphoric Acid. $H_4P_2O_7$, c.w. 178.

1. $AgNO_3$ produces a white precipitate of $Ag_4P_2O_7$, soluble in HNO_3, and in $(NH_4)HO$.

2. Albumen gives no precipitate.

Metaphosphoric Acid. HPO_3, c.w. 80.

1. $AgNO_3$ produces a white gelatinous pr[illegible]te of $AgPO_3$.

2. Albumen produces a flocculent white precipitate when added to metaphosphoric acid, and the same precipitate when added to a solution of a metaphosphate acidified with acetic acid.

3. $MgSO_4 + NH_4Cl + (NH_4)HO$ produces no precipitate.

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* If the test be applied to an acid solution of a phosphate insoluble in water (*e.g.* $Ca_3(PO_4)_2$ in HCl), the free acid is first nearly neutralised with $(NH_4)HO$, sodium acetate next added, and then Fe_2Cl_6; after this the mixture is boiled. The precipitate, which is of a reddish brown colour, contains all the iron and phosphoric acid: the filtrate contains the base. The phosphoric acid can easily be separated from the iron, and obtained as a soluble ammonium salt by treating the ferric phosphate with $(NH_4)_2S$.

99. BORIC ACID. $B(HO)_3$, c.w. 62.

1. $BaCl_2$ produces a white precipitate of $Ba(BO_2)_2$, soluble in acids.

2. $AgNO_3$ produces in strong solutions a yellowish white precipitate. In dilute solutions Ag_2O is precipitated.

3. H_2SO_4 or HCl, added to hot concentrated solutions of alkaline borates, produces on cooling a crystalline precipitate of $B(HO)_3$.

*4. If alcohol containing free boric acid be kindled, it burns with a green flame, best seen on stirring the mixture. Borates may be examined in this way by first adding strong H_2SO_4, to liberate the $B(HO)_3$.

5. If the solution of a borate be made distinctly acid with HCl, and turmeric paper dipped into it, the latter on gentle warming acquires a brown tint, which is turned blue by caustic soda.

6. Moistened with H_2SO_4 and heated in the lamp flame, a green coloration is produced.

100. OXALIC ACID. $C_2H_2O_4$, c.w. 90.

1. $BaCl_2$ produces in neutral solutions a white precipitate of C_2O_4Ba, soluble in HNO_3, in HCl, and in $C_2H_2O_4$.

2. $AgNO_3$ produces a white precipitate of $C_2O_4Ag_2$, soluble in HNO_3, and in $(NH_4)HO$.

3. $CaCl_2$ produces even in dilute solutions a white precipitate of C_2O_4Ca, soluble in HNO_3, and in HCl, but nearly insoluble in $(NH_4)HO$, and in acetic acid. On igniting C_2O_4Ca, a white residue of $CaCO_3$ is left, which effervesces on the addition of an acid.

*4. Heated with strong H_2SO_4, effervescence takes

* To a suspected solution containing free boric acid add NH_4HO + $MgSO_4$ and boil down to dryness, then add strong H_2SO_4 and alcohol nd ignite if boric acid present have green fl

place from the escape of a mixture of CO and CO_2, and the former may be kindled at the mouth of the test tube, and will burn with a pale blue flame. *No blackening* of the mixture occurs as in the case of organic acids, which yield CO on heating with H_2SO_4.

101. HYDROFLUORIC ACID. HF, c.w. 20.

1. $BaCl_2$ produces a white precipitate of BaF_2, soluble in HCl, and sparingly in NH_4Cl.

2. $CaCl_2$ produces a gelatinous and almost transparent precipitate of CaF_2, difficult to discern in the fluid, but made more apparent on addition of $(NH_4)HO$. The precipitate is very difficultly soluble in HCl, even on boiling, and is nearly insoluble in acetic acid.

*3. Heated with H_2SO_4, all fluorides are decomposed with evolution of HF, which is recognised by its power of etching glass. A *very characteristic* oily appearance is noticed whenever a fluoride is warmed with H_2SO_4 in a test tube. (See **47**.)

The etching is best done by placing the fluoride in a platinum crucible, or small leaden cup, along with strong H_2SO_4, and covering the mouth with a waxed watch-glass, convex side downwards, on which a few scratches have been made with a needle. The concave side of the watch-glass is filled with water to prevent the wax on the other side from melting, and the crucible or cup is then gently heated. On removing the glass and melting off the wax by gentle warming, the glass will be found etched at the unprotected parts.*

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* If the fluoride contain much silica, SiF_4 is evolved instead of HF, and is detected by heating the substance with H_2SO_4 in a test tube, and leading the evolved gas into water. Silica will separate out in flocculent tufts, and H_2SiF_6 will be found in solution.

4. Heated with a mixture of borax and $HKSO_4$, on a loop of platinum wire in the non-luminous gas flame, BF_3 is produced, which momentarily colours the flame green.

102. CARBONIC ACID. H_2CO_3 or $H_2O + CO_2$.

1. $BaCl_2$ produces in neutral solutions a white precipitate of $BaCO_3$, soluble in acids with effervescence.

* 2. Treated with dilute HCl, all carbonates at once evolve CO_2 with effervescence, and if this gas be conducted into lime-water, it produces a turbidity from formation of $CaCO_3$. (The experiment may be conveniently performed by placing the carbonate and dilute acid in one test tube and the lime-water in another. As soon as the CO_2 has collected, it may be decanted into the lime-water tube—care being taken to prevent any liquid from being decanted with it—and on shaking the latter the lime-water will become turbid.)

103. SILICIC ACID. $Si(HO)_4$, c.w. 96.

1. $BaCl_2$ produces a white precipitate of $SiBa_2O_4$, which is decomposed on addition of HCl, and $Si(HO)_4$ separates out as a gelatinous precipitate.

2. HCl, added drop by drop to a strong solution of a silicate, produces a gelatinous precipitate of $Si(HO)_4$, but if added to a dilute solution or in large excess, no precipitate is obtained until the mixture has been evaporated to dryness and ignited, when SiO_2 separates out, and this is not re-dissolved on addition of HCl.

3. Fused with Na_2CO_3 in a loop of platinum wire in

the non-luminous gas flame, effervescence occurs from the disengagement of CO_2, and the bead is transparent on cooling, unless the Na_2CO_3 be in excess.

* 4. Fused with microcosmic salt on a loop of platinum wire in the non-luminous gas flame, solution does not take place, but the silica floats about on the bead undissolved.

104. The remaining six acids of this group are precipitated or decomposed by one or other of the group reagents for *bases*, and are therefore precipitated in the course of examination for bases, or expelled on the addition of HCl. The action of the *base* group-reagents is as follows :—

Ag Group.	H_2SO_3 decomposed by HCl, with evolution of SO_2.
	$H_2S_2O_3$ decomposed by HCl, with evolution of SO_2 and separation of S.
As Group.	H_3AsO_3 precipitated by H_2S as As_2S_3 (yellow).
	H_3AsO_4 ,, ,, ,, ,,
	HIO_3 decomposed by H_2S, with formation of an iodide and separation of S.
Fe Group.	H_2CrO_4 precipitated by $(NH_4)_2S$ as $Cr_2(HO)_6$.

The following are additional tests for these acids :—

105. SULPHUROUS ACID. H_2SO_3, c.w. 82.

1. $BaCl_2$ produces a white precipitate of $BaSO_3$, soluble in HCl. This solution, on addition of chlorine

water, yields a white precipitate of $BaSO_4$, the sulphite being oxidized to sulphate.

2. $AgNO_3$ produces a white precipitate of Ag_2SO_3, which darkens on heating, from precipitation of Ag.

* 3. Added to a mixture of zinc and HCl, H_2S is produced and recognized by its smell and blackening action on paper moistened with solution of a lead salt.

4. H_2S decomposes free H_2SO_3 with separation of sulphur.

106. THIOSULPHURIC (formerly called Hyposulphurous) ACID. $H_2S_2O_3$, c.w. 114.

1. $BaCl_2$ produces a white precipitate of BaS_2O_3, soluble in HCl, with formation of sulphur as a yellow precipitate.

*2. HCl, or H_2SO_4, produces no immediate precipitate, but on standing a short time sulphur is precipitated (yellow), and simultaneously SO_2 is evolved.

3. $AgNO_3$ produces a white precipitate of $Ag_2S_2O_3$, which rapidly darkens in colour and becomes ultimately black from formation of Ag_2S. These changes are hastened by heat.

4. Fe_2Cl_6 produces a reddish coloration, but on heating it is decolorized, the ferric being reduced to ferrous chloride.

107. ARSENIOUS ACID. H_3AsO_3, c.w 126.

*1. $AgNO_3$ produces in *neutral* solutions a yellow precipitate of Ag_3AsO_3, soluble in $(NH_4)HO$. If no precipitate appear at first owing to the solution not being neutral, add a few drops of a *very* dilute solution of $(NH_4)HO$ until it appears. (See **75**, 2.)

2. $MgSO_4 + NH_4Cl + (NH_4)HO$ produce no precipitate.

(See also reactions for Arsenic, **75.**)

108. ARSENIC ACID. H_3AsO_4, c.w. 142.

* 1. $AgNO_3$ produces in neutral solutions a light brown precipitate of Ag_3AsO_4. If necessary, add very dilute ammonia, as in the preceding case.

2. $MgSO_4 + NH_4Cl + (NH_4)HO$ produce a white precipitate of $MgNH_4AsO_4$.

(See also reactions for Arsenic, **75.**)

109. IODIC ACID. HIO_3, c.w. 176.

1. $BaCl_2$ produces a white precipitate of $Ba(IO_3)_2$, soluble in HNO_3.

2. $AgNO_3$ produces a white crystalline precipitate of $AgIO_3$, readily soluble in $(NH_4)HO$, but sparingly soluble in HNO_3.

3. SO_2 produces at first a precipitate of I, which is converted into HI on addition of excess of the re-agent.

* 4. On heating, iodates are decomposed, oxygen being evolved, and in some cases iodine is also given off in violet vapours.

110. CHROMIC ACID. H_2CrO_4, c.w. 118·2.

1. $BaCl_2$ produces a yellow precipitate of $BaCrO_4$, soluble in HCl and HNO_3, but insoluble in acetic acid.

2. H_2S in presence of HCl reduces the solution to Cr_2Cl_6 (green), with separation of S. In neutral solutions, $Cr_2(HO)_6$ is precipitated along with S.

3. SO_2 reduces solutions of chromates to the chromic salt, the colour of which is green. Chromates are likewise reduced by zinc and a dilute acid, by oxalic acid and dilute sulphuric acid, by strong H_2SO_4, by strong HCl, and by boiling the solution acidified with HCl or H_2SO_4 along with alcohol.

4. $AgNO_3$ produces a dark red precipitate of Ag_2CrO_4, soluble in HNO_3 and in $(NH_4)HO$.

*5. Lead acetate produces a bright yellow precipitate of $PbCrO_4$, soluble in NaHO, but soluble with difficulty in dilute HNO_3.

(See also reactions for Chromium, **83.**)

Reactions of the Acids belonging to Group III.

111. Acids precipitated by $AgNO_3$, and not by $BaCl_2$.

Hydrochloric, Hydrobromic, Hydriodic, Hydrocyanic, Hypochlorous, Nitrous, and Hydrosulphuric Acids.

HYDROCHLORIC ACID. HCl, c.w. 36·5.

1. $AgNO_3$ produces a white curdy precipitate of AgCl, which becomes violet on exposure to light. The precipitate is insoluble in HNO_3, but soluble in $(NH_4)HO$, in KCN, in $Na_2S_2O_3$, and also to some extent in NaCl.

* **2.** Heated with H_2SO_4 and MnO_2, chlorides yield chlorine gas, recognized by its smell, bleaching action, and green colour.

3. *Dry* chlorides, when heated in a retort with H_2SO_4 and $K_2Cr_2O_7$, yield CrO_2Cl_2 (chromium oxychloride), which distils over into the receiver as a dark red liquid, decomposed by addition of water or $(NH_4)HO$, yielding a yellow solution, which, on addition of a lead salt, gives a yellow precipitate of $PbCrO_4$.

112. HYDROBROMIC ACID. HBr, c.w. 81.

1. $AgNO_3$ produces a pale yellow precipitate of AgBr, insoluble in dilute HNO_3, soluble in strong $(NH_4)HO$, and readily in KCN and $Na_2S_2O_3$.

* 2. Chlorine passed through a solution of a bromide decomposes it with liberation of Br, which dissolves in the liquid and colours it yellow. If this solution be shaken up with ether, the bromine is dissolved by it, and the yellow ethereal solution floats above the liquid which becomes colourless. If the ethereal solution be then separated from the liquid, and NaHO be added, the yellow colour disappears, and NaBr and $NaBrO_3$ are produced. On evaporation and ignition, oxygen is evolved and NaBr alone remains, which may be tested as in 3.

3. Heated with H_2SO_4 and MnO_2, bromides yield red vapours of Br, recognized by its powerful odour.

4. Heated in a retort with $K_2Cr_2O_7$ and H_2SO_4, dry bromides yield dark red vapours, which condense in the receiver to a liquid of the same colour, which

consists of pure bromine, and is decolorized on adding excess of $(NH_4)HO$. (Compare Hydrochloric Acid test, **111,** 3.)

113. HYDRIODIC ACID. HI, c.w. 128.

1. $AgNO_3$ produces a pale yellow precipitate of AgI, insoluble in dilute HNO_3, and very difficultly soluble in $(NH_4)HO$, but readily in KCN and $Na_2S_2O_3$.

2. Cuprous sulphate * produces a dirty-white precipitate of Cu_2I_2, which separates most completely if the solution be made slightly alkaline with Na_2CO_3. The reagent produces no precipitate in solutions of chlorides or bromides.

3. KNO_2 produces no reaction in solutions of iodides until a few drops of HCl or H_2SO_4 are added, when iodine is at once liberated and colours the solution yellow. If a little starch solution be now added, a deep blue coloration results from the formation of starch iodide. On warming the blue liquid the colour disappears, but reappears on cooling. The production of blue starch iodide is the most characteristic test for iodine.

* 4. Chlorine water (or the gas) liberates iodine from iodides, but *excess* of Cl causes the formation of ICl_3, which is colourless, and gives no blue coloration with starch solution. If therefore chlorine water be added drop by drop to a solution of an iodide mixed with starch solution, a blue coloration is produced,

which disappears on further addition of the reagent.

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* Prepared by dissolving a mixture of two parts $CuSO_4$ and five parts $FeSO_4$ in water, or by the action of SO_2 on $CuSO_4$.

5. Free iodine (liberated by either of the above methods) is dissolved by CS_2, forming a violet-coloured solution. If then, a solution of iodine be shaken up with CS_2, the latter acquires a violet colour. Chloroform may be substituted for CS_2.

6. Heated with MnO_2 and dilute H_2SO_4, violet vapours of iodine are obtained, which colour paper moistened with starch, blue.

114. HYDROCYANIC ACID. HCN, C.W. 27.

1. $AgNO_3$ produces a white precipitate of AgCN, insoluble in HNO_3, with difficulty in $(NH_4)HO$, but readily in KCN and $Na_2S_2O_3$. AgCN is decomposed on ignition, and metallic Ag remains; this serves to distinguish it from AgCl, which is not decomposed on ignition.

2. If a solution of $FeSO_4$, which has become oxidized by exposure to the air, be added to the solution of a cyanide made alkaline with NaHO, a bluish green precipitate is formed, which is a mixture of Prussian blue with the hydrated oxides of iron. On adding HCl, these last are dissolved, and the blue precipitate remains.

* 3. HCl decomposes nearly all cyanides with evolution of HCN, recognized by its odour, resembling bitter almonds. If a cyanide be thus decomposed in a small porcelain basin, covered by a similar basin on which a drop of $(NH_4)_2S_2$ (yellow) adheres, the

latter is converted into $(NH_4)CNS$, which gives a blood-red coloration on addition of Fe_2Cl_6 and HCl.

NOTE.—$Hg(CN)_2$ cannot be detected by the above methods. The dry substance is detected by igniting in a small tube, when cyanogen gas is evolved, or the solution is decomposed by H_2S and filtered from the HgS; the filtrate contains HCN.

115. HYPOCHLOROUS ACID. HClO, c.w. 52·5.

1. $AgNO_3$ produces a white precipitate of AgCl.

2. $Pb(NO_3)_2$ produces a white precipitate, which changes in colour to red, and ultimately to brown from formation of PbO_2.

3. $MnCl_2$ produces a dark brown precipitate of $MnO(HO)_2$.

4. Indigo and litmus solution are decolorized, especially on addition of an acid.

*5. Dilute acids decompose hypochlorites with evolution of Cl. HNO_3 evolves HClO from hypochlorites.

116. NITROUS ACID. HNO_2, c.w. 47.

1. $AgNO_3$ produces a white precipitate of $AgNO_2$, soluble in a large excess of water.

2. H_2S, in presence of acid, produces a precipitate of S, and $(NH_4)NO_3$ remains in solution.

* 3. $FeSO_4$, in presence of an acid, produces a black coloration from solution of NO in the $FeSO_4$.

(See also **113**, test 3.)

117. HYDROSULPHURIC ACID (Sulphuretted Hydrogen). H_2S, c.w. 34.

1. $AgNO_3$ produces a black precipitate of Ag_2S, insoluble in dilute acids.

2. Lead acetate, even when highly dilute, produces a black precipitate of PbS.

3. Sodium nitro-prusside, in presence of NaHO, produces a reddish violet coloration, even in very dilute solutions. The colour disappears in a short time.

*4. HCl or H_2SO_4 decomposes most sulphides with evolution of H_2S, recognized by its disagreeable odour and by its blackening paper moistened with solution of lead.

Reactions of the Acids belonging to Group IV.

118. Acids not precipitated by any reagent.

Nitric, Chloric, and Perchloric Acids.

NITRIC ACID. HNO_3, c.w. 63.

1. Nitrates when heated evolve oxygen, and in some cases nitrous vapours also. On fusing a nitrate and adding a fragment of charcoal, vivid deflagration occurs.

(For the reactions of Nitric Acid, see **22.**)

119. CHLORIC ACID. $HClO_3$, c.w. 84·5.

1. H_2SO_4 decomposes chlorates with evolution of Cl_2O_4, a greenish yellow gas, having a powerful odour

Dissolve $FeSO_4$ in strong H_2SO_4 and pour carefully on to this the nitrate. a brown ring will be formed where two meet-

If heated, violent explosions occur: the mixture ought therefore to be kept cold, and only *very* small quantities should be used.

2. When chlorates are heated, oxygen is evolved and a metallic chloride remains, which may be dissolved in water and precipitated, as AgCl, by $AgNO_3$. (See **5**.)

3. Chlorates are reduced by SO_2 with liberation of chlorine or its oxides, hence if the solution of a chlorate be coloured blue with indigo solution, it is decolorized on adding H_2SO_4 and solution of Na_2SO_3. (Distinction from perchlorates).

*4. HCl decomposes chlorates with evolution of Cl and Cl_2O_4, a mixture called *euchlorine.*

5. Heated with charcoal, chlorates deflagrate violently.

PERCHLORIC ACID. $HClO_4$, c.w. 100·5.

1. H_2SO_4 does not act upon perchlorates in the cold, and on heating white fumes of $HClO_4$ are given off, but no explosions occur. (Compare **119**, 1.)

*2. KCl produces in strong solutions a white precipitate of $KClO_4$.

3. Indigo solution is not decolorized when added to perchlorates warmed with HCl, as euchlorine is not evolved.

4. Dry perchlorates evolve oxygen on heating.

5. Anhydrous perchloric acid is a colourless fuming liquid, which explodes with great violence when dropped on wood charcoal. (*Roscoe.*)

6. Perchlorates are not reduced by SO_2. (Compare **119**, 3.)

TABLE J.

120. Detection of Inorganic Acids in Mixtures.

[The following acids are found in the examination for bases, which ought always to *precede* the examination for acids :—H_2SO_3, $H_2S_2O_3$, H_2CO_3, H_2S, $Si(HO)_4$, H_2CrO_4, H_3AsO_3, and H_3AsO_4.

(a) Acids in Soluble Bodies.

1. Neutralize a portion of the solution with $(NH_4)HO$ and add $BaCl_2$ (or $Ba(NO_3)_2$ if Ag, Hg_2, or Pb be present): precipitate indicates H_2SO_4 H_3PO_4, H_3AsO_3, H_3AsO_4, $Si(HO)_4$, H_2CrO_4, $\overline{Ox}$, $\overline{Ci}$, $\overline{T}$,* and large quantities of $B(HO)_3$ and HF.

To precipitate, add water and then HCl: if a precipitate remain, $\mathbf{H_2SO_4}$ was present.

2. To another portion of the neutralized solution add $AgNO_3$: a precipitate indicates one or more of the following acids :—

(*a*) HCl, HBr, HI, HCN, $H_4Fe(CN)_6$, $H_3Fe(CN)_6$, H_2S.

(*b*) H_3PO_4, H_3AsO_4. H_3AsO_3, H_2CrO_4, $Si(HO)_4$, $B(HO)_3$, $\overline{Ox}$, $\overline{T}$, and $\overline{Ci}$. To the precipitate add cold dilute HNO_3. Acids under (*a*) are insoluble, those under (*b*) soluble.

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* $\overline{Ox}$, $\overline{Ci}$, and $\overline{T}$ are contractions for oxalic, citric, and tartaric acids (For the further separation of these organic acids, see Table K.)

DETECTION OF ACIDS UNDER (*a*).

To a portion of the solution add starch paste and one drop of a solution of N_2O_3 in H_2SO_4. Blue coloration indicates **HI**. Add now chlorine water till the blue colour disappears, and shake with chloroform. If this becomes reddish brown in colour, the presence of **HBr** is indicated. **HCl** is detected in the presence of the others by boiling down the solution to dryness and distilling the residue with $K_2Cr_2O_7$ and H_2SO_4. See also note, page 117.

DETECTION OF ACIDS UNDER (*b*).

Test separately for each acid by the methods already given.

SEPARATION OF H_3AsO_3, H_3AsO_4, AND H_3PO_4.

Acidify solution with HCl, add Na_2SO_3, and heat until no smell of SO_2 is given off. Pass H_2S through the hot solution, filter, and test for H_3PO_4 with ammonium molybdate: yellow precipitate indicates **H_3PO_4**. Precipitate another portion with magnesia mixture, and test both precipitate and filtrate for arsenic.

TEST FOR THE REMAINING ACIDS BY THE FOLLOWING REACTIONS GIVEN UNDER EACH ACID.

For HCN by test 3, **114.**

For $H_4Fe(CN)_6$ by tests 3 and 4, **126.**

For $H_3Fe(CN)_6$, by tests 2 and 3, **127.**

For H_2S, by test 4, **117.**

*For HNO_3, by tests *b* and *c*, **22.**

For $HClO_3$, by tests 1 and 2, **119.**

For $\overline{Ox}$ and HF, by $CaCl_2$ + acetic acid. Confirm $\overline{Ox}$ by test 4, **100,** and HF by test 3, **101.**

For $B(HO)_3$ by tests 4 and 5, **99.**

For $Si(HO)_4$, by tests 2 and 4, **103.**

For H_2CrO_4, by tests 4 and 5, **110.**

For H_2SO_3 by test 3, **105,** and smell of SO_2 on adding HCl.

For CO_2 by test 2, **102.**

For $H_2S_2O_3$ by tests 2 and 3, **106.**

(β) Acids in Insoluble Bodies.

Some idea of the nature of the compound is generally obtained by the preliminary examination (Tables A and B). If not dissolved by the ordinary reagents, the substance must be fused with about four times its weight of a mixture of Na_2CO_3 and K_2CO_3. When cold, extract the fused mass with water, and filter if necessary. The filtrate contains the acid, and is neutralized with HCl or HNO_3, and examined by the methods given under (*a*).

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* If HI be present, it must first be removed by addition of $FeSO_4$ + $CuSO_4$. (See **113,** 2.)

The sulphates of barium, strontium, and calcium are decomposed by boiling with a *concentrated* solution of Na_2CO_3. Filter, and examine the filtrate for the acid.

Nitric acid and aqua regia oxidize sulphides to sulphates: hence the solution of a sulphide in these acids always contains H_2SO_4. In such cases a separate portion of the substance must be examined for H_2SO_4 by boiling with HCl, diluting with water, and then testing with $BaCl_2$.

NOTE.—In mixtures of chlorides, bromides, and iodides, or any two of them, proceed as follows. Place a small quantity of the mixture in a test tube, add water and a few pieces of MnO_2 * (free from chlorides), then *one drop only* of dilute sulphuric acid and boil: violet vapour indicates **Iodides.** Add another drop of the dilute acid and boil again. Proceed in this way till no more violet vapour is given off. Then add about 2 c.c. dilute sulphuric acid and boil: brown vapour indicates **Bromides.** Boil till all bromine is expelled, allow to cool completely, add to the residue an equal bulk of strong sulphuric acid and warm: a green gas indicates **Chlorides.** Confirm by observing if a piece of moistened red blotting-paper held in the mouth of the tube is bleached.

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* Powdered MnO_2 produces too much "bumping" to be used for this purpose.

121. GROUPING OF THE ORGANIC ACIDS.

GROUP I. (TARTARIC ACID GROUP).

Group reagent, $CaCl_2$.

Tartaric, Citric, and Malic Acids (Oxalic Acid, see 100).

Acids which are precipitated by $CaCl_2$ in the cold or on boiling.

GROUP II. (BENZOIC ACID GROUP).

Group reagent, Fe_2Cl_6.

Benzoic and Succinic Acids.

Acids which are not precipitated by $CaCl_2$, but which give precipitates with Fe_2Cl_6 in neutral solutions.

GROUP III.

Group reagent, $AgNO_3$.

Ferro-cyanic, Ferri-cyanic, Sulpho-cyanic, Acetic and Formic Acids.

Acids precipitated by $AgNO_3$ in neutral solutions, and not by $CaCl_2$, or Fe_2Cl_6. Acetates and Formates are only precipitated in concentrated solutions.

Reactions of the Organic Acids belonging to Group I. (Tartaric Acid Group.)

Acids precipitated by $CaCl_2$ in the cold or on boiling.

Tartaric and Citric Acids.

122. TARTARIC ACID. $C_4H_6O_6$.

1. $CaCl_2$ in neutral solutions produces a white precipitate of $C_4H_4CaO_6$, soluble in acids, and in ammoniacal salts. The precipitate is soluble in KHO, but is re-precipitated when the solution is boiled, and on cooling is re-dissolved.

*2. KCl produces in solutions containing $\overline{T}$ in excess a white crystalline precipitate of $C_4H_5KO_6$, soluble in mineral acids and alkalies, insoluble in acetic acid. The precipitation is promoted by stirring, or by addition of alcohol.

3. Lime-water produces in neutral solutions a white precipitate $C_4H_4CaO_6$ (flocculent at first, afterwards crystalline), soluble in tartaric acid and NH_4Cl, but re-precipitated in crystals from these solutions after standing some time.

4. Add to some calcium tartrate which has been washed two or three times by decantation (after pouring off the wash water as completely as possible), a drop or two of $(NH_4)HO$ and a crystal of $AgNO_3$, and heat the mixture in a test tube. A lustrous mirror of silver will deposit on the tube.

5. Heated with strong H_2SO_4, the mixture darkens

rapidly from separation of carbon, and SO_2, CO, and CO_2 are evolved.

6. Heated to redness, the substance darkens in colour and gives off the characteristic odour of burnt sugar.

123. CITRIC ACID. $C_6H_8O_7$.

1. $CaCl_2$ produces no precipitate in neutral solutions in the cold, but on boiling, $Ca_3(C_6H_5O_7)_2$ is precipitated, and is not soluble in KHO, but soluble in $(NH_4)HO$.

* 2. Lime-water produces no precipitate in cold neutral solutions, but on boiling, $Ca_3(C_6H_5O_7)_2$ is precipitated.

3. $AgNO_3$ produces in neutral solutions a white flocculent precipitate of $C_6H_5Ag_3O_7$, soluble in $(NH_4)HO$: this solution does not blacken on boiling.

4. Heated with strong H_2SO_4, CO_2 and CO are evolved without any darkening in colour; on continued heating, however, the mixture darkens, and SO_2 is evolved.

5. Heated to redness, irritating fumes are given off, readily distinguished from those given off by heating the preceding acid.

123a. MALIC ACID. $C_4H_6O_5$.

1. $CaCl_2$ produces no precipitate in neutral solutions in the cold, but upon boiling, $C_4H_4CaO_5$ separates from strong solutions. The precipitate when heated with $(NH_4)HO$ and $AgNO_3$ causes no separation of silver.

2. Lime-water does not precipitate solutions of malic acid or of malates even on boiling. (Compare **123**, 2.)

* 3. $AgNO_3$ produces in neutral solutions a white granular precipitate of $C_4H_4Ag_2O_5$, which becomes grey on boiling.

4. Heated with strong H_2SO_4, CO_2 and CO are evolved, the fluid then darkens and SO_2 is evolved.

Reactions of the Acids belonging to Group II. (Benzonic Acid Group.)

124. Acids precipitated by Fe_2Cl_6, and not by $CaCl_2$.

Benzoic and Succinic Acid.

BENZOIC ACID. $C_7H_6O_2$.

1. Fe_2Cl_6 produces, in neutral solutions, a buff-coloured precipitate of ferric benzoate, decomposed by $(NH_4)HO$ with formation of a more basic benzoate and ammonium benzoate. Ferric benzoate is soluble in HCl with liberation of benzoic acid.

2. Heated with H_2SO_4 benzoic acid does not blacken.

*3. Heated in an open tube, the acid sublimes in needle-shaped crystals, and an irritating vapour is given off. When kindled, the crystals burn with a smoky flame.

125. SUCCINIC ACID. $C_4H_6O_4$.

1. Fe_2Cl_6 produces, in neutral solutions, a reddish brown bulky precipitate of ferric succinate, soluble in mineral acids, and decomposed by $(NH_4)HO$ in a similar manner to ferric benzoate.

2. Lead acetate produces a white precipitate of lead succinate, soluble in excess of the reagent and in HNO_3.

* 3. $BaCl_2$, in presence of $(NH_4)HO$ and alcohol, produces a white precipitate of barium succinate; this reaction serves to distinguish this acid from benzoic, which does not give a similar precipitate.

4. Heated in an open tube, the acid sublimes in fine needles; and when kindled, the crystals burn with a bluish but not smoky flame.

Reactions of the Acids belonging to Group III

126. Acids precipitated by $AgNO_3$, in strong neutral solutions.

Ferro-cyanic, Ferri-cyanic, Sulpho-cyanic, Acetic, and Formic Acids.

FERRO-CYANIC ACID. $H_4Fe(CN)_6$.

1. $AgNO_3$ produces a white precipitate of $Ag_4Fe(CN)_6$, insoluble in dilute HNO_3 and in $(NH_4)HO$, but soluble in KCN.

*2. $CuSO_4$ produces a reddish brown precipitate of $Cu_2Fe(CN)_6$.

3. Fe_2Cl_6 produces a deep blue precipitate of Prussian blue, insoluble in dilute mineral acids, soluble in oxalic acid, and decomposed by NaHO with separation of $Fe_2(HO)_6$.

4. $FeSO_4$ gives a light blue precipitate, which rapidly darkens in colour by oxidation.

127. FERRI-CYANIC ACID. $H_3Fe(CN)_6$.

1. $AgNO_3$ produces an orange-coloured precipitate of $Ag_3Fe(CN)_6$, insoluble in dilute HNO_3, soluble in $(NH_4)HO$ and KCN.

*2. $FeSO_4$ produces a blue precipitate of $Fe_3Fe_2(CN)_{12}$ (Turnbull's blue), insoluble in acids, but decomposed by alkalies.

3. Fe_2Cl_6 produces no precipitate, but alters the colour to a greenish brown.

127a. SULPHO-CYANIC ACID. H(CN)S.

1. $AgNO_3$ produces a white curdy precipitate of Ag(CN)S insoluble in dilute acids but soluble in $(NH_4)HO$, and in solution of K(CN)S. From the solution in $(NH_4)HO$ it crystalizes in shining scales.

2. $CuSO_4$ produces in strong solutions a black crystalline precipitate of $Cu(CN)_2S_2$ which changes on standing to the cuprous salt $Cu_2(CN)_2S_2$ which is white. This change takes place at once by the addition of reducing agents (*e.g.* SO_2 or $FeSO_4$) to the cupric salt.

*3. Fe_2Cl_6 produces a blood-red coloration from formation of a soluble sulpho-cyanate of iron. The colour is destroyed by addition of alkalies, by $HgCl_2$ and by many acids (*e.g.* HNO_3, H_3PO_4, $H_2C_2O_4$, HIO_3), but not by HCl even when concentrated.

128. ACETIC ACID. $C_2H_4O_2$.

1. $AgNO_3$ produces, in neutral solutions, a white crystalline precipitate of $C_2H_3AgO_2$, soluble in $(NH_4)HO$ and in hot water.

2. Fe_2Cl_6, in neutral solutions, produces a deep red coloration. On boiling, all the iron separates as a light brown precipitate of basic acetate, and the fluid becomes colourless.

*3. A strong solution heated with H_2So_4 and alcohol yields acetic ether, recognized by its characteristic and pleasant odour.

129. FORMIC ACID. CH_2O_2.

1. $AgNO_3$ produces, in neutral *concentrated* solutions, a white precipitate of $CHAgO_2$, which rapidly darkens, especially on heating, owing to separation of Ag.

2. Fe_2Cl_6 produces a similar reaction with formates as with acetates.

*3. $Hg(NO_3)_2$ produces a white precipitate of the formate of mercury, which, however, soon becomes grey from separation of Hg.

*4. *Cold strong* H_2SO_4 decomposes formates with effervescence, CO being evolved. On heating, the gas comes off rapidly, and if lighted, burns with a blue flame.

130. *Higher fatty acid.*

STEARIC ACID. $C_{18}H_{36}O_2$.

1. Heated with alkalies, a soap is formed. If mutton fat (which contains the acid combined with glycerine) be heated with NaHO sodium stearate is formed, which dissolves in warm water and the glycerine separates. Soaps are decomposed by acids, which unite with the base, and the fat separates and may be dissolved in alcohol. From this solution the fat crystallizes in needles.

130a. CARBOLIC ACID OR PHENOL. $C_6H_5(HO)$.

1. Bromine water gives a yellowish white precipitate (Tribromo-phenol) even in dilute solutions.

2. Ferric chloride produces a violet coloration.

3. Heated with ammonia and a drop of sodium hypochlorite, a deep blue colour is produced, which becomes red on addition of acids.

130b. SALICYLIC ACID. $C_7H_6O_3$.

1. Heated with lime, phenol is evolved, easily recognized by its smell (like tar).

2. Ferric chloride produces even in very dilute solutions a deep violet colour, which disappears on addition of acids or alkalies.

3. Bromine water gives a white precipitate. (Compare **130a** 1.)

TESTS FOR ORGANIC ALKALOIDS AND CERTAIN OTHER ORGANIC BODIES.

131. MORPHINE. $C_{17}H_{19}NO_3$ (Opium).*

1. KHO and $(NH_4)HO$ precipitate morphia from its salts, readily soluble in excess of KHO, less readily in excess of $(NH_4)HO$.

2. Concentrated HNO_3, added to powdered morphia or its salts, produces an orange red coloration, changing afterwards to yellow. The reaction is best seen on a porcelain crucible lid. Addition of $SnCl_2$ or of $Na_2S_2O_3$ destroys the colour.

3. A neutral solution of Fe_2Cl_6 produces, when added to morphia (either in the dry state or in solution), a deep blue colour.

4. HIO_3, when added either to free or combined morphia, is decomposed with liberation of I, which coluors the liquid brown. Starched paper added to the solution becomes blue. Addition of $(NH_4)HO$ deepens the brown colour.

5. H_2SO_4 produces no coloration with morphia or its salts, but on adding a crystal of $K_2Cr_2O_7$ a bright green colour is produced.

132. MECONIC ACID, $C_7H_4O_7$ (Opium).

1. Fe_2Cl_6 produces a deep red coloration, which does not alter on boiling (like ferric acetate), nor on treatment with $HgCl_2$ (like iron sulphocyanide).

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* These names refer to the substances from which the alkaloids, &c. are derived.

2. Lead acetate produces a white precipitate of lead meconate, insoluble in acetic acid.

OPIUM SOLUTIONS :—

Opium contains both morphia and meconic acid; hence, in examining solutions of opium, the reactions of both these substances are obtained. The reaction of Fe_2Cl_6 with meconic acid is highly characteristic, and as meconic acid only occurs in opium, its detection in solutions of opium serves as an indirect indication of the presence of morphia. The two substances are separated by precipitating with lead acetate in presence of acetic acid, and filtering from the lead meconate obtained. The filtrate contains acetate of morphia. If the lead meconate be washed thoroughly and then suspended in water and H_2S passed through, PbS is precipitated, and meconic acid remains in solution and may be obtained in crystals by evaporation.

133. STRYCHNINE. $C_{21}H_{22}N_2O_2$ (Strychnos nux vomica and St. Ignatius' bean).

1. Strong pure H_2SO_4 dissolves strychnine without any discoloration of the solution, even when heated to 100° C. This solution produces certain characteristic colours, with the following reagents :—

(*a*) PbO_2, blue coloration, changing to violet, then red, and finally yellow.

(*b*) $K_2Cr_2O_7$, blue coloration, changing soon to yellowish red.

(*c*) $K_4Fe(CN)_6$, violet coloration, changing less quickly.

(*d*) MnO_2, violet coloration changing to dark red.

2. Strong HNO_3 dissolves strychnine without discoloration unless heated, when the solution becomes yellow.

3. The taste of strychnine is intensely bitter, and is perceptible even in very dilute solutions.

4. Strychnine, when taken in poisonous doses, produces tetanic convulsions. A most characteristic test, founded on this property, consists in injecting a trace of strychnine under the skin of a young frog, which is soon seized with tetanic convulsions, the body becomes rigid and arched, and the animal soon dies.

134. BRUCINE. $C_{23}H_{26}N_2O_4 + 4\ H_2O$ (Strychnos nux vomica and St. Ignatius' bean).

1. HNO_3, when added to brucine, dissolves it, and produces an intense red coloration, which becomes yellow on heating. If $SnCl_2$, $Na_2S_2O_3$ or $(NH_4)_2S$ be then added, the colour changes to violet. (Compare corresponding reaction with Morphia, **131,** 2.)

135. QUININE. $C_{20}H_{24}N_2O_2$ (Cinchona Bark).

1. Chlorine water, when added to an acid solution of quinine or its salts, produces no coloration until $(NH_4)HO$ is added, when a bright green colour is produced. If $K_4Fe(CN)_6$ be added before the $(NH_4)HO$, a red coloration is produced, soon changing to dirty brown.

2. KHO or $(NH_4)HO$ produces, in solutions of quinine salts, a white amorphous precipitate of quinine,

which, on standing, becomes crystalline. The precipitate is scarcely soluble in KHO, but slightly soluble in $(NH_4)HO$. If a mixture of a quinine solution and $(NH_4)HO$ be shaken up with ether, the precipitated quinine is dissolved. (Compare test 2 for Cinchonine, **136.**)

3. Solutions of quinine or its salts are fluorescent in a high degree, and possess an extremely bitter taste.

136. CINCHONINE. $C_{20}H_{24}N_2O$ (Cinchona Bark).

1. Chlorine water produces no coloration, even on addition of $(NH_4)HO$, which produces a yellowish precipitate.

2. KHO or $(NH_4)HO$ produces, in solutions of cinchonine salts, a white amorphous precipitate of cinchonine, insoluble in excess, and not dissolved when shaken up with ether. (Compare test 2 for Quinine, **135.**)

3. Solutions of cinchonine have a bitter taste and an alkaline reaction. The salts are less soluble in water and alcohol than those of quinine.

137. URIC ACID. $C_5H_4N_4O_3$ (Urine).

1. HNO_3 dissolves uric acid with effervescence, and the solution when evaporated to dryness and moistened with $(NH_4)HO$ assumes a bright purple colour (murexide).

2. Na_2CO_3 solution dissolves uric acid. If this solution be placed on paper moistened with $AgNO_3$ solu-

tion, a brown stain is produced, owing to the reducing action of the uric acid.

138. UREA. CH_4N_2O (Urine).

1. HNO_3, when added to solutions of urea, unites with it, forming the nitrate, which separates out in crystalline plates, which are tolerably soluble in hot water and alcohol, but nearly insoluble in HNO_3.

2. KHO added to urea decomposes it on heating, and NH_3 is evolved.

3. $Hg(NO_3)_2$ produces a white precipitate of variable composition.

4. HNO_2 decomposes urea at once into CO_2, H_2O, and N. $CH_4N_2O + 2\,HNO_2 = CO_2 + 4\,N + 3\,H_2O$.

5. When chlorine is passed into an aqueous solution of urea the following reaction takes place—$CH_4N_2O + 6\,Cl + H_2O = CO_2 + 2\,N + 6\,HCl$.

139. CHOLESTERINE. $C_{26}H_{44}O$ (Biliary Calculi).

1. KHO does not saponify cholesterine, although in many of its properties it resembles oils and fats.

2. Boiling alcohol dissolves cholesterine, and on cooling it crystallizes out in colourless plates.

140. GRAPE SUGAR. $C_6H_{12}O_6$ (Fruit, Honey, Diabetic Urine, &c.).

1. H_2SO_4 forms with grape sugar a definite compound of a yellow colour. No charring occurs as in the case of cane sugar.

2. Solution of grape sugar, when mixed with a few

drops of $CuSO_4$ and excess of KHO, produces on boiling a red precipitate (Cu_2O), caused by the reducing action of the grape sugar. Solution of cane sugar does not give this reaction until boiled with a single drop of H_2SO_4, which converts it into grape sugar.

3. An aqueous solution of grape sugar when mixed with yeast and kept at a temperature of 27° undergoes fermentation, and CO_2 is evolved. The mixture is placed in a test tube provided with a conducting tube which dips into lime water; over these a bell jar is placed (to prevent absorption of CO_2 from the air) and the apparatus kept for some hours at the necessary temperature. Turbidity in the lime water indicates the presence of grape sugar. (As CO_2 is sometimes given off by the yeast itself, it must be carefully tested before use in the manner described.)

141. ALBUMIN (White of Egg).

1. Boiling water causes albumin to coagulate and it becomes at the same time insoluble, so that if a solution in cold water be heated, coagulation at once takes place.

2. $HgCl_2$ coagulates albumin, even when present in exceedingly small quantities. Coagulation also takes place with solutions of other salts, *e.g.* $CuSO_4$, $SnCl_2$, $AgNO_3$.

3. Most acids precipitate albumin from its solutions. HNO_3 acts most readily, and is therefore used to detect the presence of dissolved albumin. Acetic,

tartaric, and ortho-phosphoric acids do not coagulate albumin, except in very concentrated solutions.

CASEIN (Milk and Cheese).

1. Casein is insoluble in water, but is kept in solution in milk by a small quantity of free alkali. Such a solution does not coagulate by heat, but a film forms on its surface when heated. If this film be removed another forms in its place.

2. All acids (except carbonic) precipitate casein from its solutions; the precipitate is soluble in excess of the reagent.

3. Infusion of rennet (the inner membrane of the stomach of a calf) coagulates casein completely.

142. STARCH. $C_6H_{10}O_5$ (Potato, Wheat, &c.). See **43**, *b*.

143. TANNIC ACID. $C_{27}H_{22}O_{17}$ (Gall-nuts).

1. Solution of gelatin (isinglass) produces a yellowish flocculent precipitate. A piece of animal membrane gives the same precipitate.

2. Fe_2Cl_6 produces a dark bluish black precipitate (ink).

144. GALLIC ACID. $C_7H_6O_5$. (Gall-nuts).

1. Solution of gelatin produces no precipitate.

2. Fe_2Cl_6 produces in neutral solutions a bluish black precipitate.

145. Detection of CARBON, HYDROGEN, NITROGEN, CHLORINE, SULPHUR AND PHOSPHORUS, in ORGANIC BODIES :—

(*a*) CARBON.—Mix the substance intimately with powdered CuO, place in a hard glass tube, fill up with small pieces of CuO, and close the tube with a cork, through which a conducting tube passes into a flask containing clear lime-water. On heating, the carbon is oxidized to CO_2, which renders the lime-water turbid. Organic liquids are examined in a similar way: they are placed in a small bulb tube which is placed inside a hard glass tube and gently heated as soon as the CuO in front is red-hot. In this way the vapour passes over the CuO, and CO_2 is produced.

(*b*) HYDROGEN.—Proceed as in *a* (taking care to use perfectly dry CuO), but connect the conducting tube with a weighed $CaCl_2$ tube. Heat the hard glass tube as before, and then weigh the $CaCl_2$ tube. Gain in weight indicates that water has been formed by the oxidation of the hydrogen in the substance. When much water is present, it is visible in the bulb of the $CaCl_2$ tube.

(*c*) NITROGEN.—Mix the substance intimately with powdered soda-lime, and observe if NH_3 is given off on heating. Ascertain this by the smell and by the action on red litmus paper.

(*d*) CHLORINE.—Mix the substance intimately with quick-lime (free from chlorine), place the mixture in a test tube, and heat to redness. Extract the residue with water, filter, and add solution of $AgNO_3$ to the filtrate. By this means organic chlorine compounds, which are not precipitated by solution of $AgNO_3$, are decomposed, the chlorine uniting with the calcium to form $CaCl_2$.

(*e*) SULPHUR.—Solid compounds may be tested by fusing them with a mixture of pure solid KHO and KNO_3. The fused mass when cold is dissolved in water, acidified with HCl and tested in the usual way with solution of $BaCl_2$.

Liquids are gently heated with strong HNO_3, or with a mixture of HCl and $HClO_3$, the solution is then diluted and tested with $BaCl_2$.

(*f*) PHOSPHORUS.—Fuse the substance as in (*e*) with a mixture of KHO and KNO_3, or heat it with strong HNO_3. Add water and test as in **98**, 2, 5, or 6.

Phosphorus may also be detected by slightly charring the substance and proceeding as in **60.**

146. Detection of METALS in PRESENCE OF ORGANIC MATTER :—

The reactions given for the various metals are not reliable in presence of organic matter, and as metals such as arsenic, lead, mercury, &c., in cases of poisoning have frequently to be detected when mixed with organic matter, it is necessary to state the methods which are used for this purpose.

METHOD A.—By means of dialysis.

Add a little arsenious acid or some corrosive sublimate to some minced meat (to represent the organic matter, such as stomach, liver, &c., of an actual poisoning case), stir this in about 200 c.c. of distilled water and add a little pure hydrochloric acid. Place this mixture on a dialyser and float it in a litre of water contained in a porcelain basin. After the lapse of twenty-four hours concentrate the contents of the basin on a water-bath, precipitate the arsenic or mercury by sulphuretted hydrogen, and confirm their presence by the tests already given.

This method may be applied to the separation from organic matter of any crystalloid substance whether inorganic or organic.

METHOD B.—By destroying the organic matter.

Place a similar mixture of meat with some metallic poison in a porcelain basin, add an equal bulk of strong pure hydrochloric acid, and warm on the water-bath, adding from time to time a few crystals of potassium chlorate and replacing the water removed by evaporation. Continue in this way until the organic matter has completely disappeared, heat to expel chlorine, filter, pass sulphuretted hydrogen through the warm solution, and test the precipitate by the reactions previously given.

METHOD C.—Separation of arsenic from organic matter.

Place a mixture of meat and arsenious acid in a glass retort, add some rock salt and pure sulphuric acid in quantity insufficient to decompose all the salt. Heat the retort and collect the distillate in a receiver kept very cold. The arsenic present is converted by this treatment into trichloride, which being volatile passes into the receiver, and may be precipitated with sulphuretted hydrogen or recognised by other tests for arsenic.

TABLE K.

146. Separation of Organic Acids.

Remove the metals of the Silver, Copper, and Iron Groups by their respective group reagents. Neutralize the solution with $(NH_4)HO$, add $CaCl_2$, shake well, and allow to stand for at least twenty minutes.

PRECIPITATE. | FILTRATE.

Oxalic and Tartaric Acids.
Wash with water, warm precipitate with acetic acid, and filter.

RESIDUE. | FILTRATE.

CaŌx.
Confirm by tests 3 & 4, **100,** to ascertain the presence of **Oxalic Acid.**

CaT̄.
Evaporate first to dryness and confirm by tests 4 & 5, **122,** to ascertain the presence of **Tartaric Acid.**

Remaining Acids.
Add to filtrate 3 volumes of strong alcohol and filter.

PRECIPITATE | FILTRATE.

Citric, Malic and Succinic Acids.
Wash with alcohol, dissolve in HCl, add $(NH_4)HO$, boil and filter.

PRECIPITATE. | FILTRATE.

CaC̄i.
Confirm by dissolving again in HCl, and adding $(NH_4)HO$. Confirm by tests 4 and 5, **123,** to ascertain the presence of **Citric Acid.**

Malic and succinic acids. Add alcohol, filter, wash with a little alcohol, dissolve residue in HNO_3 and evaporate to dryness on the water bath. Add excess of Na_2CO_3 solution, boil, filter, neutralize exactly with HCl, heat and add solution of $CaSO_4$ to a portion. A white precipitate indicates **Malic Acid.** To the remaining portion add $CaCl_2$ in excess, filter, and add alcohol to the filtrate. A gelatinous precipitate indicates **Succinic Acid.**

Benzoic Acid, &c.
Heat to expel alcohol, neutralize *exactly* with HCl, and add Fe_2Cl_6. Filter.

PRECIPITATE. | FILTRATE.

Ferric Benzoate.
Wash the precipitate, warm with $(NH)_4HO$, filter, evaporate filtrate nearly to dryness. Add HCl to obtain shining crystals of **Benzoic Acid.** Confirm by test 3, **124.**

Acetic and Formic Acid, &c., &c.

(The filtrate is coloured red in presence of acetic or formic acid.) See Note.

NOTE.—Acetic and formic acids are tested for in the original solution. Neutralize (if necessary) with Na_2CO_3, boil down to dryness, and test a portion for **Acetic Acid** by ether test **128,** 3. Or distil with H_2SO_4 and digest distillate with excess of PbO to obtain basic acetate of lead. To another portion of dry residue add dilute H_2SO_4 and distil. Heat part of the distillate with PbO to obtain crystals of lead formiate. To another part add $(NH_4)HO$ till neutral, and then $AgNO_3$ (test 1, **129**), to ascertain the presence of **Formic Acid.**

Uric (**137**), tannic, and gallic acids are separately tested. Gallic and tannic are separated by isinglass, or by dialysis through a small piece of bladder (**143** and **144**).

PART V.

REACTIONS OF THE RARE METALS.

147. THE following rare metals are considered here by themselves, and their position in the various groups indicated. For their separation from each other and from the commonly occurring metals, a larger manual must be consulted.

Tungsten, Thallium, Palladium, Rhodium, Osmium, Ruthenium, Gold, Platinum, Iridium, Molybdenum, Selenium,* Tellurium, Uranium, Indium, Beryllium, Zirconium, Cerium, Lanthanum, Didymium, Titanium, Tantallum, Vanadium, Lithium, Cæsium, Rubidium.

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* Selenium, although a metalloid, is conveniently included here.

Grouping of the Rare Metals.

148. PRECIPITATED IN SILVER GROUP.

Tungsten (as Tungstic Acid), Thallium (as Chloride).

PRECIPITATED IN COPPER GROUP.

Palladium, Rhodium, Osmium, Ruthenium.

PRECIPITATED IN ARSENIC GROUP.

Gold, Platinum, Iridium, Molybdenum, Tellurium, Selenium.

PRECIPITATED IN IRON GROUP.

Uranium, Indium (Thallium), *as sulphides;* Beryllium, Zirconium, Cerium, Lanthanum, Didymium, Titanium, Tantalum, *as hydrated oxides.*

PRECIPITATED AS SULPHIDES ON ADDING HCl TO THE FILTRATE FROM THE IRON GROUP.

Vanadium (Tungsten).

FOUND IN POTASSIUM GROUP.

Lithium, Cæsium, Rubidium.

Reactions of the Rare Metals of the Silver Group.

149. TUNGSTEN. W, c.w. 184.

1. HCl produces a white precipitate of Tungstic acid H_2WO_4, insoluble in excess, but soluble in $(NH_4)HO$. H_2SO_4 or HNO_3 produces the same precipitate.

2. $(NH_4)_2S$ does not precipitate tungstates of the alkalies, until an acid be added, when tungsten trisulphide WS_3 is precipitated as a light brown powder.

3. $K_4Fe(CN)_6$ in acid solutions yields a reddish brown coloration, and on standing, a precipitate of the same colour.

4. $SnCl_2$ produces a yellow precipitate. On adding HCl and heating, the precipitate becomes blue.

5. Zn in presence of phosphoric acid produces a bright blue colour.

6. With microcosmic salt in the reducing flame a blue bead is obtained, which changes to red on heating with $FeSO_4$.

150. THALLIUM. Tl, c.w. 203·5.

1. HCl in strong solutions produces a white precipitate of TlCl, soluble in a large quantity of water.

2. $(NH_4)_2S$ produces a black precipitate of Tl_2S, insoluble in $(NH_4)HO$, but soluble in HCl, H_2SO_4, and HNO_3.

3. KI precipitates Tl I (yellow) even in dilute solutions.

4. $PtCl_4$ precipitates a double chloride of thallium and potassium Tl_2PtCl_6 of an orange colour.

5 Zn added to thallium solutions precipitates the metal.

6. Thallium salts colour the lamp flame intensely green.

Reactions of the Rare Metals of the Copper Group.

151. PALLADIUM. Pd, c.w. 106·6.

1. H_2S precipitates PdS as a black powder, insoluble in $(NH_4)_2S$, and soluble in boiling HCl and aqua regia.

2. $Hg(CN)_2$ precipitates palladium cyanide, $Pd(CN)_2$, yellowish white and gelatinous, soluble in $(NH_4)HO$ and in HCl.

3. KI produces a black precipitate of PdI_2.

4. KCl in strong solutions precipitates K_2PdCl_6 in yellow needles, insoluble in absolute alcohol, but soluble in water, forming a dark-red fluid.

152. RHODIUM. Rh, c.w. 104·2.

1. H_2S precipitates Rh_2S_3 (brown), especially in warm solutions; it is insoluble in $(NH_4)_2S$, but soluble in hot HNO_3.

2. KHO produces a yellowish precipitate of $Rh(HO)_3$ H_2O, soluble in excess of the reagent; on boiling the solution, brown $Rh(HO)_3$ is precipitated.

3. Zn produces a precipitate of metallic rhodium.

4. Dry compounds when ignited in a current of hydrogen are reduced to metal, insoluble in aqua regia, but soluble on fusing with $HKSO_4$.

153. OSMIUM Os, c.w. 199·1.

1. H_2S in presence of acid precipitates OsS (brownish black), insoluble in $(NH_4)_2S$.

2. OsO_4 (osmic acid) decolorizes indigo solution.

3. KI is decomposed with liberation of iodine.

4. Na_2SO_3 yields a violet coloration, and after a time $OsSO_3$ (deep blue) separates out.

5. $FeSO_4$ precipitates black OsO_2.

6. Zn in presence of acids precipitates the metal.

7. Dry compounds when ignited in a current of hydrogen are reduced to metal.

8. The metal and the mono- and di-oxides, when heated in air, yield OsO_4, which is recognized by its peculiar irritating smell (resembling chlorine). This is an exceedingly characteristic reaction.

154. RUTHENIUM. Ru, c.w. 104·4.

1. H_2S produces no immediate precipitate, but on standing, the solution turns blue, and brown Ru_2S_3 is precipitated.

2. $(NH_4)_2S$ precipitates Ru_2S_3, difficultly soluble in excess.

3. KHO precipitates black sesquioxide $Ru(HO)_3$, insoluble in excess, but soluble in acids.

4. KCNS in pure solutions produces on standing, a red coloration, which becomes first purple, and then on heating violet.

5. Zn produces a blue coloration, and ultimately the metal separates out.

Reactions of the Rare Metals of the Arsenic Group

155. GOLD. Au, c.w. 197.

1. H_2S produces in cold solutions black Au_2S_3, in hot solutions brown Au_2S. Both precipitates are insoluble in HNO_3 and in HCl, but dissolve in aqua regia. They dissolve in yellow, but not in colourless ammonium sulphide.

2. $(NH_4)_2S$ produces a black precipitate of Au_2S_3.

3. $(NH_4)HO$ in strong solutions precipitates ammonium aurate (fulminating gold).

4. $FeSO_4$ produces a precipitate of the metal, as a brown powder, which, when rubbed, assumes a yellow colour and metallic lustre.

5. KNO_2 also produces a precipitate of the metal.

6. $SnCl_2 + SnCl_4$ produce a purplish precipitate (purple of Cassius) even in highly dilute solutions; the precipitate is insoluble in HCl.

7. Heated on charcoal with Na_2CO_3 before the blowpipe, malleable yellow beads of the metal are obtained. (See also flame reaction, **59.**)

156. PLATINUM. Pt, c.w. 197·5.

1. H_2S produces in cold solutions on standing, brown PtS_2; on heating, the precipitate forms at once. It is soluble in a large excess of yellow ammonium sulphide; it is insoluble in HCl and in HNO_3, but soluble in aqua regia.

2. $(NH_4)_2S$ produces the same precipitate as H_2S.

3. NH_4Cl produces a yellow crystalline precipitate of $(NH_4)_2PtCl_6$, more sparingly soluble in alcohol than in water. On heating the dried precipitate, metallic platinum is left in a finely divided state.

4. KCl produces a yellow crystalline precipitate of K_2PtCl_6, also more insoluble in alcohol than in water. On heating the dried precipitate, a mixture of Pt and KCl is obtained.

5. $SnCl_2$ produces no precipitate, but, in solutions containing HCl in excess, yields a dark brown coloration due to the formation of dichloride.

6. $FeSO_4$ on long-continued boiling produces a precipitate of the metal.

7. Fused on charcoal with Na_2CO_3 before the blow-pipe flame, yields the metal as an infusible grey powder. (See also flame reaction, **59**.)

157. IRIDIUM. Ir, c.w. 198.

1. H_2S first removes the colour and sulphur separates out; afterwards brown iridium sulphide is precipitated.

2. $(NH_4)_2S$ produces the same precipitate, soluble in excess.

3. KCl produces a dark brown precipitate of K_2IrCl_6, insoluble in a concentrated solution of the reagent.

4. NH_4Cl produces a dark red precipitate of $(NH_4)_2IrCl_6$.

5. $FeSO_4$ decolorizes solutions of iridium, but produces no precipitate.

6. Zn precipitates the metal as a black powder.

158. MOLYBDENUM. Mo, c.w. 96.

1. H_2S precipitates in warm solutions, after some time, brownish black MoS_3, soluble in $(NH_4)_2S$.

2. Zn, in acid solutions, produces a blue coloration, which changes to green, and lastly to black, when Mo_2O_3 separates out.

3. KCNS, when added to an HCl solution, produces on addition of Zn a crimson coloration.

4. Na_2HPO_4 produces, in a nitric acid solution of ammonium molybdate, a yellow precipitate (in dilute solutions only after the lapse of some time). The precipitation is aided by *gentle* heating.

159. TELLURIUM. Te, c.w. 128.

1. H_2S produces, in presence of acid, a brown precipitate of TeS_2, easily soluble in $(NH_4)_2S$.

2. H_2O produces a white precipitate of H_2TeO_3.

3. KHO or Na_2CO_3, when added to an HCl solution, produces a white precipitate of H_2TeO_3, soluble in excess of either reagent.

4. Na_2SO_3 or $SnCl_2$ produces a black precipitate of the metal.

5. Zn precipitates the metal as a black powder.

6. Fused with Na_2CO_3, yields Na_2Te, which, on treatment with HCl, yields H_2Te, recognized by its very disagreeable odour.

160. SELENIUM. Se, c.w. 79·5.

1. H_2S produces, in presence of acid, a yellow precipitate of doubtful composition, which darkens in colour on heating, and is soluble in $(NH_4)_2S$.

2. $BaCl_2$ produces a white precipitate of BaSe, soluble in HCl.

3. SO_2 and $SnCl_2$ produce in acid solutions a red precipitate of Se.

4. Heated in the reducing flame on charcoal, selenium compounds yield a highly characteristic smell, resembling that of horse-radish.

Reactions of the Rare Metals of the Iron Group.

161. URANIUM. U, c.w. 120.

1. $(NH_4)_2S$ produces a brown precipitate of oxysulphide, soluble in colourless ammonium sulphide to a black liquid.

2. Alkalies produce a yellow precipitate of hydrated oxide, insoluble in excess of the reagent.

3. $(NH_4)_2CO_3$ produces a precipitate of the double carbonate of uranium and ammonium, soluble in excess of the reagent.

4. $BaCO_3$ produces a precipitate of hydrated oxide in cold solutions.

5. $K_4Fe(CN)_6$ produces a reddish brown precipitate.

6. Fused with borax in the reducing flame, uranium compounds yield pale green beads.

162. INDIUM. In, c.w. 113·4.

1. $(NH_4)_2S$, in presence of $(NH_4)HO$ and tartaric acid, produces a white precipitate, which turns yellow on addition of acetic acid.

2. Alkalies produce a precipitate of the hydrate, insoluble in excess of the reagent.

3. Alkaline carbonates precipitate the carbonate, soluble in excess of $(NH_4)_2CO_3$, but not in excess of K_2CO_3 or Na_2CO_3.

4. $BaCO_3$ precipitates solutions of indium completely.

5. $K_4Fe(CN)_6$ produces a white precipitate.

6. Zn precipitates the metal in shining plates.

7. Heated with Na_2CO_3 in the reducing blow-pipe flame, metallic beads are obtained.

8. Indium compounds impart a bluish violet tinge to the flame.

163. BERYLLIUM. Be, c.w. 9·3.

1. $(NH_4)_2S$ precipitates the hydrate (flocculent) like alumina in appearance, but differs from it in dissolving on continued boiling with NH_4Cl. Beryllium chloride is formed and ammonia is driven off.

2. KHO precipitates the hydrate, soluble in excess. If this solution be diluted and well boiled, the beryllium hydrate is re-precipitated.

3. Na_2CO_3 precipitates the carbonate (white), soluble in a large excess.

4. $(NH_4)_2CO_3$ precipitates the carbonate (white), easily soluble in excess of the reagent.

5. $BaCO_3$ in cold solutions precipitates the beryllium completely.

164. ZIRCONIUM. Zr, c.w. 89·6.

1. $(NH_4)_2S$ produces a precipitate of hydrate, in-

soluble in excess. KHO and $(NH_4)HO$ produce the same precipitate, insoluble in excess of these reagents.

2. Alkaline carbonates precipitate the carbonate as a flocculent powder, soluble in a large excess of K_2CO_3 and in a small excess of $(NH_4)_2CO_3$. From this latter solution the hydrate is re-precipitated on boiling.

3. $BaCO_3$ does not precipitate zirconium completely, even on boiling.

165. CERIUM. Ce, c.w. 92·2.

1. $(NH_4)HO$ produces a precipitate of a basic salt, insoluble in excess.

2. KHO produces a precipitate of the white hydrate, insoluble in excess of the reagent. The precipitate becomes yellow on exposure to the air.

3. $(NH_4)_2CO_3$ produces a white precipitate, soluble in excess of the reagent.

4. $C_2H_2O_4$ precipitates cerous oxalate, insoluble in excess, but soluble in HCl.

5. $BaCO_3$ precipitates cerium completely after the lapse of some time.

6. Chlorine, passed through a solution mixed with sodium acetate, precipitates the peroxide (light yellow).

166. LANTHANUM. La, c.w. 92·9.

1. $(NH_4)HO$ produces a precipitate of a basic salt.

2. KHO precipitates the hydrate (white), which does not alter on exposure to air.

3. $(NH_4)_2CO_3$ produces a precipitate, insoluble in excess of the reagent.

4. $C_2H_2O_4$ precipitates the oxalate, insoluble in excess, but soluble in HCl.

167. DIDYMIUM. D, c.w. 95.

1. $(NH_4)HO$ produces a precipitate of a basic salt, insoluble in excess of the reagent, but sparingly soluble in NH_4Cl.

2. KHO precipitates the hydrate (white), which does not alter on exposure to air.

3. $(NH_4)_2CO_3$ produces a white precipitate, insoluble in excess of the reagent, but soluble in NH_4Cl.

4. $C_2H_2O_4$ produces an almost complete precipitation of the oxalate, soluble in hot HCl.

5. Fused with microcosmic salt in the reducing flame, a reddish violet bead is obtained.

6. Fused with carbonate of soda in the oxidizing flame, greyish white beads are obtained.

168. TITANIUM. Ti, c.w. 50.

1. $(NH_4)HO$ or $(NH_4)_2S$ precipitates H_2TiO_3 (titanic acid), insoluble in excess of either reagent.

2. KHO also precipitates H_2TiO_3, insoluble in excess.

3. $BaCO_3$ produces the same precipitate.

4. $Na_2S_2O_3$ when boiled with titanium solutions precipitates them completely.

5. Zn produces, first a blue coloration, then a blue precipitate, which ultimately becomes white.

6 Fused with $FeSO_4$ and microcosmic salt in the reducing flame, a bright red bead is obtained.

Reactions of the Rare Metals precipitated as Sulphides on adding HCl to the filtrate from the Iron Group.

169. VANADIUM. V, c.w. 51·3.

1. H_2S produces no precipitate in acid solutions, but a blue coloration by reducing to a lower oxide.

2. $(NH_4)_2S$ produces in a solution containing H_2SO_4 a brown precipitate of the sulphide, soluble in excess of the reagent.

3. NH_4Cl in solutions of alkaline vanadates, precipitates the metal completely as ammonium metavanadate (white). This precipitate loses ammonia on heating, and leaves a residue of V_2O_5.

4. $K_4Fe(CN)_6$ produces, in acid solutions, a green precipitate, not dissolved by acids.

5. SO_2, or oxalic acid, reduces acid solutions of V_2O_5 to a lower oxide of vanadium, of a bright blue colour.

6. Zn, added to a solution of V_2O_5 in H_2SO_4 (diluted with H_2O), produces a series of changes in colour, and ultimately a violet solution is obtained, which rapidly becomes brown in air by oxidation.

Reactions of the Rare Metals found in Potassium Group.

170. LITHIUM. Li, c.w. 7.

1. $Pt.Cl_4$ produces no precipitate.

2. Na_2HPO_4 produces, on boiling the solution, a white precipitate of $2\,Li_3PO_4 + H_2O$. The precipitate

is soluble in HCl, and is not re-precipitated by $(NH_4)HO$ unless the solution be boiled.

3. Lithium compounds tinge the lamp flame bright crimson.

171. CÆSIUM. Cæ, c.w. 133.

1. $PtCl_4$ produces a crystalline light yellow precipitate of $2(CæCl) + PtCl_4$, insoluble in boiling water. (The corresponding potassium salt is dissolved by repeated treatment with boiling water.)

2. Tartaric acid produces a precipitate of the acid tartrate of cæsium, more soluble in water than the corresponding compound of rubidium.

3. Volatile cæsium salts colour the flame violet.

172. RUBIDIUM. Rb, c.w. 85·4.

1. $PtCl_4$ produces a crystalline light yellow precipitate of $2(RbCl) + PtCl_4$, insoluble in boiling water.

2. Tartaric acid produces a precipitate of the acid tartrate of rubidium, which is much more insoluble in water than the corresponding cæsium compound.

3. Volatile salts of rubidium colour the flame violet.

PART VI.

QUANTITATIVE ANALYSIS.

EXAMPLE I.

Determination of Filter Ash.

WEIGH accurately a porcelain or platinum crucible which has been heated to redness and then allowed to become *quite* cold. Select six Swedish filter-papers of uniform diameter, fold each as in the sketch, and then coil round it a thin platinum wire, leaving a loose end (A). Place the crucible on a glazed porcelain tile,

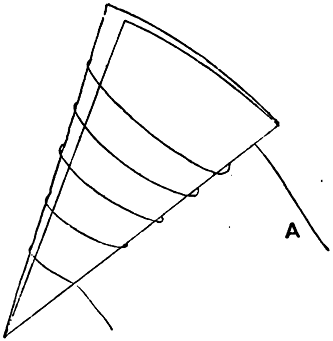

FIG. 20.

kindle the point of the paper and hold it over the crucible until the paper is reduced to ashes. Detach the ashes carefully from the wire and allow them to fall into the crucible. Repeat this with the remaining five filter-papers, and if any ash has fallen on the porcelain tile sweep it with a feather into the crucible, which is then heated to redness until the contents no longer contain any black portions.

Record the results thus :—

Crucible + ash from six filters
(diam. =) . . . =
Crucible . . . . . =

Ash from six filter-papers . . = grm.

Weight of each filter ash
(diam. =) . . . = grm.

EXAMPLE II.

Determination of Sulphuric Acid.

Weigh out about 0·2 gramme of dry powdered potassium sulphate, or any other sulphate soluble in water. This is best done by placing the salt in a dry test-tube and weighing this with its contents, then carefully shake the required quantity into a dry beaker, and re-weigh the tube. The difference between the two weighings is the weight of salt taken. To the contents of the beaker add distilled water and a few drops of pure hydrochloric acid, and heat over wire gauze with constant stirring till the liquid boils. Remove the lamp and add drop by drop clear solution of barium chloride in *very slight* excess. Heat again to the boiling-point, stirring as before ; remove the lamp and allow the precipitate to subside. To the clear liquid add a drop of barium chloride solution to see if the precipitation is complete. If no further precipitation takes place, heat again to the boiling-point for some minutes, allow to settle and then pour off the liquid through a Swedish filter-paper, leaving the precipitate in the beaker. Now add hot distilled water, one or two drops of hydrochloric acid, and boil as before, allow to settle and again pour the clear portion through the filter.* Repeat this two or three times, and finally transfer with the aid of a wash bottle every trace of the precipitate to the filter, wash repeatedly with hot distilled water until a few drops of the filtrate give no turbidity with silver nitrate, showing that the excess of barium chloride and hydrochloric acid has been removed. When the washing is complete, get the precipitate as much as possible to the point of the filter, then dry it by placing the funnel with its contents in an air-bath. When dry, transfer the precipitate as completely as possible to a weighed porcelain or platinum crucible, burn the filter (with the same precautions as in the previous example), adding the ash to the contents of the crucible, and heat to redness for some minutes, allow to become quite cold and weigh.

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* This method is called "washing by decantation."

Record the results thus :—

Tube + potassium sulphate . =
Tube − potassium sulphate . =

Potassium sulphate used . . = grm.

Crucible + barium sulphate + ash . . . . . =
Crucible . . . . . =

Barium sulphate + ash . . =
Filter ash . . . . . =

Barium sulphate . . . = grm.

Reaction:

$$K_2SO_4 + BaCl_2 = BaSO_4 + 2KCl$$
$$174{\cdot}2 + 208 = 233 + 149{\cdot}2$$

Percentage composition=	By calculation.	By analysis.	Difference.
K_2	44·89		
SO_4	55·11		
	100·00		

EXAMPLE III.

Determination of Hydrochloric Acid.

Weigh from a tube into a beaker as before about 0·1 to 0·2 gramme pure common salt or other soluble chloride, dissolve in distilled water, add a few drops of pure nitric acid and warm the solution. To the warm solution add solution of silver nitrate drop by drop until the precipitation is complete. This is easily ascertained by allowing the precipitate to subside and then adding a drop of silver nitrate solution to the clear liquid. If no further precipitation take place, cover the beaker and keep it in the dark for some hours, when the supernatant liquid should be found quite clear. Now pour the clear liquid through a filter retaining the precipitate in the beaker. Add to it hot distilled water and a drop of nitric acid and heat (stirring constantly) till it boils. Allow to subside, then pour the clear liquid through the filter as before. Repeat this process twice, then transfer the silver chloride to the filter, wash with boiling water alone, until a few drops of the filtrate give no turbidity on addition of a drop of hydrochloric acid. Finally wash the precipitate into the point of the filter, cover the funnel and place it in an air-bath. When quite dry transfer the chloride of silver *as completely as possible* to a weighed porcelain crucible, keeping the filter-paper for separate treatment. Heat the crucible gently at first, and then more strongly till the chloride just begins to fuse, allow to become perfectly cold and weigh.

Now fold the filter-paper as in the sketch, keeping the portion to which silver chloride adheres (A) in the middle of the coil, which should be as tightly rolled up as possible. Wind round it a platinum wire (previously weighed on a watch glass), and light the filter-paper, holding it over the watch glass, and allow it to burn to ashes. When it ceases to glow apply a

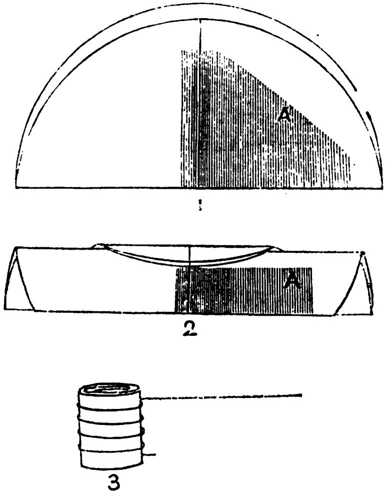

FIG. 21.

lamp flame till it is completely burned, lay it with the platinum wire on the watch glass and weigh when cold. By this process the silver chloride is reduced to metallic silver, and is weighed as such, and the quantity of silver chloride to which it corresponds is calculated. (108 parts of silver correspond to 143·5 parts of silver chloride.)

Record the results thus :—

Tube + common salt . . =
Tube – common salt . . =

Common salt used . . . = grm.

Crucible + silver chloride . =
Crucible . . . . . =

Silver chloride . . . . = grm.

Watch glass + wire + ash +
silver . . . . . =
Watch glass + platinum wire . =

Ash + silver . . . . =
Ash . . . . . . =

Silver . . . . = grm.

Silver chloride from crucible . =
Silver chloride from ash . . =

Total silver chloride . . . = grm.

Reaction:

$$\begin{array}{l} NaCl + AgNO_3 = AgCl + NaNO_3 \\ 58{\cdot}5 + 170 = 143{\cdot}5 + 85 \end{array}$$

Percentage composition=	By calculation.	By analysis.	Difference.
Na	39·32		
Cl	60·68		
	100·00		

EXAMPLE IV.

Determination of Phosphoric Acid.

Weigh out into a beaker about 0·5 gramme of sodium phosphate, ($Na_2HPO_4 + 12H_2O$), dissolve it in water, and add magnesia mixture till the precipitation is complete (add ammonia if the liquid has not a strongly ammoniacal smell) and allow to stand for twenty-four hours. Filter and wash the precipitate with water containing one-fourth of its volume of ammonia solution, until a few drops of the filtrate when neutralized with nitric acid give no precipitate on addition of silver nitrate. Dry the precipitate in the air-bath, transfer to a weighed porcelain or platinum crucible adding the filter-ash to the contents of the crucible, heat gently at first, then strongly, and weigh when cold.

Record the results as in Example II., substituting sodium phosphate for potassium sulphate, and magnesium pyrophosphate for barium sulphate.

Reaction:

$$\begin{array}{ccccccccccc} Na_2HPO_4 & + & MgSO_4 & + & (NH_4)HO & = & Mg(NH_4)PO_4 & + & Na_2SO_4 & + & H_2O. \\ 142 & + & 120 & + & 35 & = & 137 & + & 142 & + & 18 \end{array}$$

On ignition the ammonium-magnesium phosphate is converted into magnesium pyrophosphate, $Mg_2P_2O_7$.

EXAMPLE V.

Determination of Carbonic Anhydride.

Weigh out from one to two grammes of Iceland spar (calcium carbonate), place it in the flask A (Fig. 22) along with a little water, then insert a small test-tube containing hydrochloric acid in such a way that it is supported by the side of the flask and without risk of the acid escaping till required to do so. Place strong sulphuric acid in the flask B till it is nearly half full, adjust the stoppers in each flask, and close the opening C with a piece of india-rubber tubing and a small piece of glass rod. Now weigh the double flasks accurately, and incline the apparatus so that a little of the hydrochloric acid in the test-tube mingles with the water and Iceland spar in A. Carbonic anhydride is at once evolved and is freed from moisture by passing through the sulphuric acid in B. When the evolution of gas slackens, allow more acid to escape from the tube, and continue this until the carbonate is completely dissolved. Then heat the flask A with a small flame so as to expel the carbonic anhydride dissolved by the liquid; remove the stopper at C. attach an india-rubber tube to the exit tube of B and slowly draw air by suction through the apparatus until every trace of carbonic anhydride is removed. When the apparatus is quite cold replace the stopper at C and again weigh. The loss in weight represents the carbonic anhydride originally present in the spar.

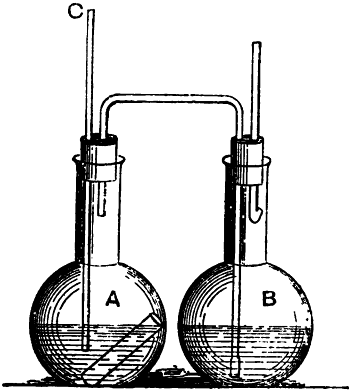

FIG. 22.

Record the results thus:—

Tube + Iceland spar . . =
Tube . . . . =
Iceland spar used . . . . = grm.

Carbonic anhydride apparatus . =
" after experiment . =
Carbonic anhydride . . . = grm.

Reaction :

$$CaCO_3 + 2HCl = CaCl_2 + CO_2 + H_2O$$
$$100 + 73 = 111 + 44 + 18$$

Percentage composition =	By calculation.	By analysis.	Difference.
CaO	56		
CO_2	44		
	100		

EXAMPLE VI.

Determination of Iron.

Weigh from a tube into a beaker about 0·2 gramme of ferrous ammonium sulphate* ($FeSO_4 + (NH_4)_2SO_4 + 6H_2O$), which has been previously powdered and dried by pressing between filter-paper. Dissolve the salt in water, add hydrochloric acid and a few drops of nitric acid (to convert the ferrous into ferric salt) and heat till it nearly boils. If sufficient nitric acid has been added the colour of the solution ought to change to brownish red. Allow to cool, add ammonia in slight excess, heat until the liquid nearly boils stirring constantly and allow to settle. Pour off the clear hot liquid through a filter and repeatedly wash by decantation, allowing the precipitate to subside each time. Then transfer the precipitate to the filter, wash thoroughly with boiling water and dry in an air-bath. Transfer the precipitate as completely as possible to a weighed crucible, add the filter ash to its contents, and heat to redness for some time. Allow to cool and weigh the iron sesquioxide (Fe_2O_3). [The student will be able to judge from the previous examples how to record the results.]

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* This salt contains 1/7th of its weight of iron.

EXAMPLE VII.

Determination of Calcium.

Weigh out about 0·2 gramme of Iceland spar and dissolve it in dilute hydrochloric acid in a beaker covered all the time with a watch glass, to prevent loss by spirting. When completely dissolved, wash any liquid adhering to the watch glass into the beaker, add ammonia in slight excess, warm the solution, and to the hot liquid add solution of ammonium oxalate until complete precipitation is effected. Now add ammonia until the fluid smells of it, cover the beaker and leave it in a warm place for at least twelve hours. Pour the clear liquid through a filter, leaving the precipitate in the beaker, wash it several times with boiling water and finally transfer it to the filter. Any precipitate adhering to the beaker is removed by rubbing with a glass rod tipped with india-rubber. Dry the precipitate in the air-bath, transfer to a platinum crucible (to which the filter ash is added) and heat at first gently over the Bunsen lamp, finally to a red heat. Now heat the crucible over a Bunsen blowpipe flame for a considerable time, and repeat this until the crucible ceases to lose weight. This serves to convert the calcium oxalate into oxide (CaO), from the amount of which the percentage of calcium in the spar is calculated.

EXAMPLE VIII.

Determination of Copper.

Weigh out about 0·2 gramme of crystallized copper sulphate ($CuSO_4 + 5H_2O$), into a porcelain basin, dissolve in water, and heat till nearly boiling. Add now pure solution of potash drop by drop until no further precipitation occurs, and continue the heating for some little time, but without allowing the liquid to boil. When the precipitate has subsided, pour the clear colourless liquid through a filter, wash three times by decantation using boiling water each time, then collect on the filter, wash thoroughly with hot water and dry in the air-bath. Detach the dried precipitate as completely as possible from the paper, place it in a weighed porcelain crucible, and burn the filter with the same precautions as with silver chloride (page 155), add the ash to the contents of the crucible and moisten it with one or two drops of nitric acid. Carefully expel the excess of acid by gentle heating, and ignite until the copper nitrate formed is converted into oxide. When quite cold, weigh, and calculate the amount of copper in the salt from the weight of copper oxide found.

EXAMPLE IX.

Preparation of Potassium Permanganate Solution for Determination of Iron, &c.

Place between 5 and 6 grammes of powdered potassium permanganate in a glass-stoppered bottle capable of holding rather more than a litre. Add now a litre of water, and shake the bottle until the salt is *completely* dissolved. When this is the case add the solution to a glass-stoppered burette, filling up to the top and then running out the solution till the liquid stands *exactly* at the uppermost line marked 0. Weigh accurately about 0·5 gramme of dry powdered ferrous ammonium sulphate ($FeSO_4(NH_4)_2SO_4+6H_2O$); place it in a beaker of about half a litre capacity, and dissolve (without the aid of heat) in about 200 c.c. of water. When dissolved add dilute sulphuric acid, place the beaker on a white porcelain tile and run in the permanganate solution from the burette, stirring constantly until the solution is distinctly pink coloured. When the reaction is seen to be nearly over, the permanganate must be added drop by drop. Now read off the point at which the permanganate stands in the burette and record the result. Weigh out a second portion of the double sulphate and repeat the process until you obtain three or four closely agreeing experiments.

Record the results thus :

Exp. I.	gramme. double salt required	c.c. permanganate.
,, II.	,, ,, ,,	c.c. ,,
,, III.	,, ,, ,,	c.c. ,,
,, IV.	,, ,, ,,	c.c. ,,

Therefore 100 c.c. permanganate = grm. double salt.

[As the salt contains one-seventh of its weight of iron, the results are easily calculated for metallic iron.]

Reaction :

$$2KMnO_4 + 10FeSO_4 + 8H_2SO_4 =$$
$$2MnSO_4 + 5Fe_2(SO_4)_3 + K_2SO_4 + 8H_2O$$

EXAMPLE X.

Determination of Iron (*Volumetric*).

Weigh out about 0·1 gramme of clean iron wire and dissolve it with the aid of heat in dilute sulphuric acid in a flask through which a current of carbonic anhydride is continually passed. When complete solution has been effected, allow to cool in the current of carbonic anhydride, dilute with water, and triturate with permanganate solution as in the previous experiment. In exact experiments a correction must be made for the impurities in the wire, which usually amount to 0·4 per cent., in other words every decigramme of iron wire corresponds to only 0·0996 gramme pure iron. Repeat this experiment two or three times and compare the results with those obtained in Example IX.

EXAMPLE XI.

Determination of Oxalic Acid.

Weigh out between 1 and 2 decigrammes of pure crystallized oxalic acid ($C_2H_2O_4 + 2H_2O$), place in a beaker with about 200 c.c. of water, add dilute sulphuric acid and warm gently. Add permanganate from the burette, stirring constantly, until the pink colour is permanent even on warming the solution. Repeat the experiment two or three times and compare the results with the preceding ones. Every 112 parts of iron as ferrous salt correspond to 126 parts of crystallized oxalic acid.

$$2KMnO_4 + 5C_2H_2O_4 + 3H_2SO_4 =$$
$$K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O.$$

EXAMPLE XII.

Preparation of Standard Oxalic Acid.

Weigh out exactly 63 grammes of pure crystallized oxalic acid. This is best done by accurately weighing a large watch glass, adding 63 grammes to the weights, and then oxalic acid to the watch glass until the balance is again in equilibrium. With the aid of a dry funnel transfer the whole of the crystals to a litre flask, wash the watch glass and funnel, and nearly fill with distilled water. Dissolve by shaking the flask, and when quite dissolved fill up exactly to the mark on the neck, and shake again thoroughly. Test the strength by means of the standard permanganate solution previously made, using for this purpose about 10 or 20 c.c. of the oxalic acid solution. Now weigh out in the manner already described, 21·3714 grammes of pure recently ignited sodium carbonate, transfer it to a quarter-litre flask, dissolve in water, fill up to the mark and shake thoroughly. Pour this solution into a burette and allow 20 c.c. (= 1 gramme Na_2O), to flow out into a porcelain basin containing about 100 c.c. water. Add a few drops of neutral litmus solution, and then the standard solution of oxalic acid until the blue colour disappears. Heat the solution to the boiling-point, again add standard acid till the colour changes from purple to bright red, and repeat this until the latter colour is constant. Repeat the experiment with another 20 c.c. of sodium carbonate solution, and if the two experiments agree, compare them with the previous ones in which the strength was estimated by means of permanganate.

Use the standard acid for determining the amount of alkali in accurately weighed quantities of caustic soda, caustic potash, soda ash, &c., &c.

EXAMPLE XIII.

Preparation of Standard Sulphuric Acid.

Measure out a litre of water and place it in a stoppered bottle, then run into it from a burette 34 c.c. of strong pure sulphuric acid, add 20 c.c. of water, shake thoroughly, and allow to become quite cold. Fill a burette with the diluted acid and ascertain, just as in the previous example, how much of it is required to saturate 20 c.c. of the sodium carbonate solution prepared in XII. From this calculate the weight of sulphuric anhydride in the litre. Take now 20 c.c. of the acid and determine the amount of sulphuric acid by precipitation with barium chloride as in II. Compare the two results, and if they agree well, take the mean as the quantity of sulphuric acid present in the solution, which must now be diluted so as to contain exactly 40 grammes SO_3 in the litre. The amount of water to be added is easily calculated. Thus if a litre is found to contain 41·6 grammes SO_3, instead of 40, then by the proportion 40 : 41·6 : : 1000 = 1040, it is necessary to add 40 c.c. water to each litre of the acid.

Use the standard acid so prepared for determining the amount of alkali in soda ash, &c.

EXAMPLE XIV.

Preparation of Standard Soda.

Dilute a solution of pure soda until the specific gravity as indicated by the hydrometer is about 1·05, and place it in a burette. Now add 50 c.c. of the standard acid (XIII.) to about 100 c.c. of water contained in a basin, add a few drops of litmus solution, and then run in from the burette caustic soda solution until the colour just changes to blue. The solution is required of such a strength that 1 c.c. acid is exactly neutralized by 1 c.c. soda. By proceeding as above the soda will be stronger than the acid, and must be diluted with the proper quantity of water. If, for example, 46 c.c. of soda are equal to 50 c.c. of acid, then 4 c.c. of water must be added to every 46 c.c. of the solution.

By means of the standard soda ascertain the amount of acid in accurately weighed quantities of acetic acid, sulphuric acid, potassium bisulphate, &c.

EXAMPLE XV.

Determination of Chlorine (*Volumetric*).

Weigh out accurately 2·3944 grammes of pure silver nitrate and dissolve in one litre of distilled water. (This is a convenient strength for the determination of the amount of chlorides in water, for other purposes a stronger solution is employed.) Place the silver solution in a burette and allow it to run drop by drop into 50 c.c. of ordinary water contained in a porcelain basin, and to which one or two drops of a solution of pure potassium chromate have been added. When the liquid becomes faintly red in colour read off the amount of silver nitrate used; the number of cubic centimetres used indicates the amount of chlorine in 100,000 parts of the water examined. The potassium chromate serves to indicate when the precipitation of silver chloride is complete, since *none* of the red silver chromate is formed until *all* of the highly insoluble silver chloride is precipitated.

EXAMPLE XVI.

Estimation of Chlorine in Bleaching Powder.

Weigh out exactly 4·95 grammes of pure dry arsenic trioxide, place it in a flask with some water, heat gently, and add pure crystallized sodium carbonate from time to time until complete solution has been effected. Allow to cool, transfer carefully to a litre flask, and fill up to the mark on the neck. This forms a deci-normal solution of arsenious acid, and may be used for the determination of iodine, chlorine, bleaching powder, &c.

Reactions :

$$As_2O_3 + 4I + 2H_2O = 4HI + As_2O_5.$$
$$As_2O_3 + Ca(ClO)_2 = CaCl_2 + As_2O_5.$$

Weigh out exactly 3·55 grammes of bleaching powder into a mortar, add water, and thoroughly mix with the aid of the pestle. Decant off the turbid fluid into a litre flask, leaving the sediment behind. Add more water to this, triturate again, transfer as before to the litre flask, and repeat this until all the bleaching powder has been transferred to the litre flask, then fill up to the mark. Shake thoroughly, and before allowing it to settle withdraw 100 c.c. by means of a pipette, and place it in a beaker. Add now from a burette the standard arsenic solution, stirring constantly, and testing from time to time (with the aid of a glass rod) the action of the fluid on strips of paper moistened with a mixture of potassium iodide and starch solution. At first the prepared paper is deeply marked blue, but as the arsenious acid is added the marks become fainter and fainter, and it is easy to determine the exact point when the paper ceases to be tinged. Read off from the burette the number of cubic centimetres of arsenic solution used; this number indicates at once the percentage of chlorine in the sample of bleaching powder.

EXAMPLE XVII.

Determination of Manganese Dioxide.

Weigh accurately from 3 to 4 grammes of dry, finely-powdered pyrolusite, place it in the flask A, Fig. 22 (see page 159), add a little water, and about 6 grammes neutral sodium oxalate. Put about 50 c.c. strong sulphuric acid in the flask B, adjust the stoppers, and weigh the entire apparatus on an accurate balance. Now attach an india-rubber tube to the exit tube of B and gently suck air from A, so as to cause sulphuric acid to pass over from B into A. As soon as this is done an evolution of carbonic anhydride begins; the gas is dried by passing through the acid in B, and escapes by the exit tube. When the evolution of gas slackens repeat the suction, and continue in this way until every trace of pyrolusite has been dissolved. When this is the case draw more acid over into A, so as to heat the liquid and expel any dissolved carbonic anhydride, then remove the stopper at c and draw air through the apparatus to replace the carbonic anhydride. When perfectly cold weigh the apparatus again; the loss in weight indicates the carbonic anhydride expelled, from the amount of which the quantity of manganese dioxide in the pyrolusite may be calculated.

Reaction:

$$\begin{array}{ccccccccccccccc} MnO_2 & + & 2H_2SO_4 & + & Na_2C_2O_4 & = & MnSO_4 & + & Na_2SO_4 & + & 2CO_2 & + & 2H_2O. \\ 87 & + & 196 & + & 134 & = & 151 & + & 142 & + & 88 & + & 36. \end{array}$$

EXAMPLE XVIII.

Determination of Lead.

Weigh out about 0·5 gramme of re-crystallized lead nitrate, dissolve in water, add ammonium carbonate in slight excess and a few drops of ammonia, heat gently, and after some time, filter. Wash the precipitate with distilled water and dry in the water-bath. Transfer the precipitate *as completely as possible* to a weighed porcelain crucible, ignite gently at first and then strongly, allow to cool and weigh the lead oxide (PbO) remaining. The filter must be burned separately as in the case of silver, and when *thoroughly ignited* may be added to the contents of the crucible.

EXAMPLE XIX.

Determination of Zinc.

Weigh out about 1 gramme of zinc sulphate crystals ($ZnSO_4 + 7 H_2O$), dissolve in water and heat nearly to the boiling-point, add by degrees solution of sodium carbonate till there is a slight excess, and then heat to the boiling-point. Allow to settle, decant the clear liquid, boil the residue with more water two or three times, then transfer to the filter, wash thoroughly, and dry the precipitate. Transfer the dry precipitate as completely as possible to a crucible, add to it the ashes of the filter-paper, heat to redness, and when cold weigh the residue of zinc oxide (ZnO.)

When these examples have been completed, the student may practise other determinations, using the same reactions. Thus, by weighing out quantities of barium chloride, silver nitrate, and magnesium sulphate, and precipitating their solutions with sulphuric acid, hydrochloric acid and sodium phosphate respectively, the methods described in II., III., and IV. may be used for the determination of barium, silver, and magnesium.

APPENDIX A.

TABLE OF THE ELEMENTS, WITH THEIR SYMBOLS AND COMBINING WEIGHTS.

Element.	Symbol.	Combining Weight.
Aluminium	Al	27·3
Antimony	Sb	120
Arsenic	As	75·2
Barium	Ba	137
Beryllium	Be	9·3
Bismuth	Bi	210
Boron	B	11
Bromine	Br	80
Cadmium	Cd	112
Cæsium	Cs	133
Calcium	Ca	40
Carbon	C	12
Cerium	Ce	92·2
Chlorine	Cl	35·5
Chromium	Cr	52
Cobalt	Co	58·7
Copper	Cu	63·5
Didymium	D	95
Erbium	E	112·6

Element.	Symbol.	Combining Weight.
Fluorine	F	19
Gold	Au	197
Hydrogen	H	1
Indium	In	113·4
Iodine	I	127
Iridium	Ir	198
Iron	Fe	56
Lanthanum	La	92·9
Lead	Pb	207
Lithium	Li	7
Magnesium	Mg	24
Manganese	Mn	55
Mercury	Hg	200
Molybdenum	Mo	96
Nickel	Ni	58·7
Niobium	Nb	94
Nitrogen	N	14
Osmium	Os	199·1
Oxygen	O	16
Palladium	Pd	106·6
Phosphorus	P	31
Platinum	Pt	197·5
Potassium	K	39·1
Rhodium	Rh	104·2
Rubidium	Rb	85·4
Ruthenium	Ru	104·4
Selenium	Se	79·5
Silver	Ag	108

Element.	Symbol.	Combining Weight.
Silicon	Si	28
Sodium	Na	23
Strontium	Sr	87·5
Sulphur	S	32
Tantalum	Ta	182·3
Tellurium	Te	128
Thallium	Tl	203·5
Thorium	Th	115·7
Tin	Sn	118
Titanium	Ti	50
Tungsten	W	184
Uranium	U	120
Vanadium	V	51·3
Yttrium	Y	61·6
Zinc	Zn	65·2
Zirconium	Zr	89·6

APPENDIX B.

WEIGHTS AND MEASURES.

Measures of length.

1 metre = 10 decimetres (dcm.).
1 metre = 100 centimetres (cm.).
1 metre = 1000 millimetres (mm.).

1000 metres = 1 kilometre.
100 metres = 1 hectometre.
10 metres = 1 decametre.

1 inch = 2·539954 centimetres.
1 foot = 3·0479449 decimetres.
1 yard = 0·91438348 metre.
1 mile = 1·6093149 kilometre.

Measures of weight.

1 gram = 10 decigrams.
1 gram = 100 centigrams.
1 gram = 1000 milligrams.

1000 grams = 1 kilogram.
100 grams = 1 hectogram.
10 grams = 1 decagram.

1 grain = 0·06479895 gram.
1 ounce (Troy.) = 31·103496 grams.
1 pound (Avd.) = 0·45359265 kilog.
1 cwt. = 50·80237689 kilogs.

Measures of capacity.

1 litre = 1 cubic decimetre.
1 litre = 1000 cubic centimetres.
1000 litres = 1 kilolitre or stere.

1 cub. in. = 16·3861759 cubic centimetres.
1 cub. ft. = 28·3153119 cubic decimetres.
1 gallon (70,000 grains.) = 4·543457969 litres

APPENDIX C

TREATMENT OF SILVER RESIDUES.

SILVER residues generally consist chiefly of silver chloride, and may be treated as follows. Add hydrochloric acid in slight excess to precipitate any soluble silver salt that may be present, and allow the precipitate to subside. Pour off the supernatant liquid, and wash the precipitate several times (by decantation) with water. Place the precipitate in an evaporating basin, add solution of caustic soda and one or two pieces of sugar, and boil the mixture, stirring constantly. As soon as the precipitate settles rapidly to the bottom, leaving the liquid clear, the heating is stopped, and the precipitate washed with common water (by decantation) till no longer alkaline, and finally once or twice with distilled water. The moist precipitate is then dissolved in pure nitric acid, and evaporated to obtain crystals of silver nitrate. If any choloride has escaped reduction, the solution in nitric acid will not be clear, in this case it must be diluted and filtered. When the quantity of silver residues is small, it is better to dissolve the finely divided silver in the smallest quantity of nitric acid possible, dilute with distilled water, and filter the solution.

TABLE

IRON GROUP IN PRESENCE OF PHOSPHORIC

Manganese, Zinc, Chromium, Barium, Strontium, and Mag-

To the filtrate from the sulphides of the Cu and As groups add $(NH_4)HO$ (till
some time and filter. Wash well with water containing $(NH_4)_2S$, and finall

RESIDUE.

NiS, CoS, Si, O_2, (CaF_2).*

Wash and test a portion with microcosmic salt. A white powder remaining undissolved indicates **Silicic Acid;** the bead becoming blue indicates **Cobalt.** Examine the remainder of the precipitate for Ni, as in Table F.

* As CaF_2 is sparingly soluble in HCl, it may be found in this residue. Its presence is detected by incinerating a portion of the residue, and heating it with strong H_2SO_4. Evolution of HF indicates **Calcium Fluoride.**

METHOD I. Cr IS ABSENT.

Boil down with a few drops of HNO_3 (yellow coloration indicates the presence of Iron). Add dilute H_2SO_4, boi
again and filter.

RESIDUE.

$BaSO_4$, $SrSO_4$, $(CaSO_4)$. Proceed as in Table G. III.

FILTRATE.

Add equal bulk of Alcoho
and filter.

RESIDUE.

$CaSO_4$

Wash, dissolve in water, add $(NH_4)HO$ and $(NH_4)_2C_2O_4$. White precipitate indicates **Calcium.** Confirm by flame reaction.

FILTRATE.

Evaporate to a small bulk, dilute with water, cautiously add Fe_2Cl_6 (until a drop of the solution gives a buff-coloured precipitate with $(NH_4)HO$, neutralize with Na_2CO_3, add a few crystals of sodium acetate and a few drops of acetic acid, boil for 15 minutes, and filter the hot liquid.

RESIDUE.

Wash with boiling water, boil precipitate with Na_2CO_3, filter, acidify the filtrate with HCl, add $(NH_4)HO$ till alkaline, white precipitate indicates **Aluminium.**

FILTRATE.

Mn, Zn, Mg (traces of **Co** and **Ni**). Add $(NH_4)HO$, NH_4Cl, and $(NH_4)_2S$. filter.

RESIDUE.

Zn, Mn (traces of **Co** and **Ni**). Wash, dissolve in HCl, and proceed as in Table F. Method I.

FILTRATE.

Boil down to a small bulk, and add Na_2HPO_4, wh
precipitate indicates **Magnesium.**

L.

ACID, &c. **Separation of Iron, Nickel, Cobalt, Aluminium, nesium, in presence of Phosphoric, Boric, and Oxalic Acids.**

alkaline), $N H_4 Cl$ and $(N H_4)_2 S$ Warm the mixture gently in a flask, shake for once with water alone. Treat the precipitate with cold dilute H Cl, and filter.

FILTRATE.

$Cr, Al, Fe, Zn, Mn, Ca, Ba, Sr, Mg, H_3Po_4, H_3BO_3$ and $H_2C_2O_4$.

Test small portions for Cr, Fe, $H_3 P O_4$. $H_3 B O_3$ and $H_2 C_2 O_4$. Adopt Method I. if Cr be absent; Method II. if present.

METHOD II. Cr IS PRESENT.

Boil down with a little $K Cl O_3$ till it smells of chlorine. Test for Ba, Sr, and Ca in a portion of the solution, as in I. To the remainder add $Fe_2 Cl_6$, as in I; evaporate down nearly to dryness, dilute with water, add $Na_2 CO_3$, or K H O, till just neutral or slightly acid, when *perfectly cold,* add $Ba C O_3$ in slight excess, place in a small flask, close with a cork, shake well, and allow to settle. Filter.

RESIDUE.	FILTRATE.
$Fe_2 (HO)_6$, $Cr_2 (HO)_6$, $Al_2 (HO)_6$. Proceed as in Table F., Method II.	**Mn, Zn, Mg** (traces of **Co** and **Ni**). Add a few drops of H Cl, boil to expe. CO_2, add $(N H_4) H O$ (till alkaline) $N H_4 Cl$, and $(N H_4)_2 S$. Filter.

RESIDUE.	FILTRATE
Mn, Zn (traces of **Co** and **Ni**). Proceed as in Table F., Method II.	Boil with $H_2 SO_4$ to remove Ba and Sr. Filter. precipitate the Ca in the filtrate by adding $(N H_4) H O$ and $(N H_4)_2 C_2 O_4$. Filter Concentrate the filtrate, add $Na_2 H P O_4$, white precipitate indicates **Magnesium**.

APPENDIX D.

TREATMENT OF PLATINUM RESIDUES.

THESE residues generally contain the platinum as double chloride of potassium or ammonium. Any soluble platinum chloride is precipitated by addition of ammonium chloride, and the filtered precipitate is dried and ignited. The residue is then thoroughly washed with hot water, and dissolved by boiling with a mixture of three parts of hydrochloric to one of nitric acid. It is best to pour off the solution from time to time and add fresh acids. When all the platinum is dissolved the solution is evaporated nearly to dryness; hydrochloric acid is added (to expel the nitric acid), and the liquid is evaporated to complete dryness on the water bath. The residue is dissolved in water or preserved dry in bottles.

QUESTIONS AND EXERCISES.

3. (*a*) How many grams of oxygen can be obtained by heating 1,080 grams of mercuric oxide?

(*b*) Twenty-four grams of oxygen are required; how much mercuric oxide must be heated?

4. (*a*) What is left when potassium chlorate is heated?

(*b*) How many grams of potassium chlorate must be heated in order to yield 500 grams oxygen?

(*c*) How much potassium chloride will remain after heating 247 grams potassium chlorate?

5. How is potassium chlorate distinguished from potassium chloride?

6. (*a*) Describe the best means of preparing oxygen?

(*b*) The residue in a flask used to prepare oxygen, consisted of 13 grams of potassium chloride; how much chlorate had been heated, and how much oxygen evolved?

(*c*) Why is manganese dioxide mixed with potassium chlorate in preparing oxygen?

8. (*a*) What happens when a taper is burned in oxygen?

(*b*) How is the presence of carbon dioxide generally ascertained?

9. (*a*) 1·4 gram of charcoal containing 96 per cent. of carbon is burnt in oxygen; how much carbon dioxide is produced?

(*b*) Contrast the burning of a candle with the breathing of an animal in air.

10. How much oxygen is required to burn 42 grams of sulphur to sulphur dioxide?

11. How much phosphoric anhydride can be obtained by burning 2·34 grams phosphorus in oxygen, and how much phosphoric acid would be obtained by dissolving the anhydride in water?

13. (*a*) How much hydrogen can be got by dissolving 15 ounces of zinc in sulphuric acid?

(*b*) Which will yield the greater quantity of hydrogen when dissolved in acids—162 grams zinc, or 140 grams iron? Why?

14. How much water will be formed by burning all the hydrogen evolved by dissolving 3·42 grams zinc in sulphuric acid?

15. How much zinc sulphate should be got by dissolving 1 lb. zinc in sulphuric acid?

19. How is nitrogen prepared?

21. One hundred grams of potassium nitrate are heated with sulphuric acid; how much nitric acid and hydrogen potassium sulphate will be obtained?

22. What are the best tests for nitric acid?

24. (*a*) How is ammonia gas prepared, and what is the residue from its preparation?

(*b*) How much ammonium chloride must be used to obtain 291 grams ammonia gas?

25. How is ammonia tested for?

27. (*a*) How is ammonium nitrate prepared?

(*b*) How much ammonium nitrate can be got from 327 grams of nitric acid?

28. How much nitrous oxide can be obtained from 437 grams of ammonium nitrate?

29. How can nitrous oxide be distinguished from oxygen?

30. If 7 grams of copper be acted on with nitric acid, how much nitric oxide will be obtained, and how much copper nitrate will remain?

31. Mention some of the properties of nitric oxide.

32. (*a*) How is carbon dioxide prepared from marble?

(*b*) How much hydrochloric acid will be needed exactly to decompose a kilogram of marble?

33. What reaction takes place when carbon dioxide and lime-water are brought together? Give the equation.

34. (*a*) How is carbon monoxide prepared from formic acid, and from oxalic acid?

(*b*) How much carbon monoxide should be obtained from 12 grams oxalic acid?

35. What happens when a mixture of carbon monoxide and dioxide is shaken up with caustic soda?

36. (*a*) Give the equation for the preparation of chlorine from common salt, manganese dioxide, and sulphuric acid?

(*b*) What is the action of hydrochloric acid on manganese dioxide?

(*c*) How much chlorine can be got by heating 560 grams manganese dioxide with salt and sulphuric acid?

(*d*) How much can be got by heating the same quantity with hydrochloric acid?

37. (*a*) Mention the chief properties of chlorine.

(*b*) How does chlorine act as a bleaching agent?

38. How much common salt is needed to prepare 109 kilograms of hydrochloric acid?

39. (*a*) What is meant by "neutralizing" an acid solution?

(*b*) What is meant by the terms "acids," "bases," and "salts"?

40. (*a*) Give the equation for the preparation of bleaching powder from lime and chlorine.

(*b*) How much chlorine is needed to convert 5 tons of slaked lime into bleaching powder?

41. (*a*) What happens when each of the following acids is brought in contact with calcium hypochlorite, sulphuric, nitric and hydrochloric?

(*b*) How can a solution of hypochlorous acid be distinguished from a solution of chlorine?

42. (*a*) Give the equation for the preparation of iodine from potassium iodide.

(*b*) Thirty-seven grams of iodine were obtained; how much potassium iodide was used?

43. What is the best test for free iodine?

44. (*a*) How is sodium iodide prepared?

(*b*) How much sodium iodide can be got from 20 grams of iodine?

45. How much bromine can be got from 85 grams potassium bromide?

46. How much sodium, bromate and bromide, should be obtained by dissolving 97 grams bromine in caustic soda?

47. (*a*) How is hydrofluoric acid prepared?

(*b*) What is its action upon glass, and how may it be used to etch glass?

48. (*a*) What is the action of strong sulphuric acid upon copper?

(*b*) How much sulphuric acid and copper must be used to obtain a kilogram of sulphur dioxide?

(*c*) What is the action of sulphur dioxide upon nitric acid, and upon potassium chromate solution?

50. (*a*) Describe in detail how sulphuretted hydrogen gas is prepared.

(*b*) How much ferrous sulphide must be used to obtain 42 grams of the gas?

51. (*a*) How does sulphuretted hydrogen serve to divide the metals into groups?

(*b*) How could you separate by means of sulphuretted hydrogen a solution containing copper, iron, and sodium?

52. How is sodium hydrate prepared?

54. (*a*) What is meant by the terms "oxidizing" and "reducing" flames?

(*b*, How is each obtained, and for what is each used?

55. (*a*) How would you distinguish, by means of the blow-pipe, salts of the following metals—copper, cobalt, lead, zinc, antimony, aluminium, chromium, and strontium?

(*b*) What happens when mercuric chloride is heated on charcoal along with sodium bi-carbonate in the blow-pipe flame?

56. Name the substance which produces each of the following reactions :—

(*a*) Bead brittle, soluble in nitric acid, sulphide black.

(*b*) No metallic bead, but incrustation of oxide on charcoal (brown).

(*c*) Bead malleable, soluble in nitric acid; solution gives with sulphuric acid a white precipitate.

(*d*) No metallic bead; after heating, moistening with cobalt chloride and re-heating gives a pink residue.

(*e*) Red bead, soluble in nitric acid; solution becomes bright blue on adding ammonia: borax bead blue.

(*f*) No metallic bead: borax bead amethyst coloured in reducing flame.

57. (*a*) How may a Bunsen lamp flame be substituted for the flames obtained by the mouth blow-pipe?

(*b*) Which is the hottest part of the Bunsen lamp flame, and which part has most reducing power?

(*c*) How are metallic films and metallic beads obtained by Bunsen's flame reactions?

58. How can arsenic, mercury, thallium, cadmium, gold and platinum be distinguished by Bunsen's flame reactions?

59. Give Bunsen's reactions for zinc, mercury, bismuth, lead, copper, and iron.

60. Give Bunsen's tests for phosphorus and sulphur, and state the reactions involved in each.

61*a*. Six substances gave the following reactions when examined by means of Table A. Name each substance.

(*a*) Water evolved on heating (reaction acid). Substance dissolved in water, and on adding HCl a gas was evolved smelling of burning sulphur. None of the group reagents gave a precipitate. Flame coloration, yellow.

(*b*) Nitrogen tetroxide evolved on heating. Substance dissolved in water, and gave a white precipitate with hydrochloric acid, soluble in hot water.

(*c*) Oxygen evolved on heating. Substance insoluble in water, but dissolved by heating with hydrochloric acid with evolution of chlorine. No precipitate with HCl, or with $HCl + H_2S$, but on adding $(NH_4)HO + NH_4Cl + (NH_4)_2S$ a flesh-coloured precipitate was obtained.

(*d*) Carbon dioxide evolved on heating strongly. Substance insoluble in water, but on adding HCl a gas was evolved with effervescence, which rendered lime-water turbid. No precipitate with the first three group reagents, but a white one with $(NH_4) HO + NH_4 Cl + (N H_4)_2 C O_3$. Flame coloration dull red.

(*e*) White sublimate on heating. Substance dissolved in water and gave no precipitate with $BaCl_2$, but a white one with $AgNO_3$, insoluble in NHO_3. No precipitate with any of the group reagents, but gave a smell of ammonia on heating with NaHO.

(*f*) Oxygen evolved on heating. Substance soluble in water, gave no precipitate with the first three group reagents, but a white one with $(NH_4) HO + NH_4Cl + (NH_4)_2CO_3$. Flame coloration green.

61β. Six substances gave the following reaction when examined by means of Table B. Name each substance.

(*a*) White and unfused on heating. Yields when heated on charcoal with sodium carbonate, a malleable bead which leaves a black mark on paper.

(*b*) Infusible and disappears on heating. Deflagrates when heated with potassium nitrate.

(*c*) Infusible when heated. Microcosmic salt bead colourless. Blue mass after heating on charcoal, moistening with $CoCl_2$ and heating again.

(*d*) White and infusible. When heated with $H_2 SO_4$, evolves gas which etches glass.

(*e*) Infusible, but darkens in colour on heating. Fused with $NaHCO_3$, yields a brittle bead.

(*f*) Fused but not volatilized on heating. Yields malleable metallic bead when heated on charcoal with $NaHCO_3$.

62. (*a*) Give the four group reagents, and state the metals precipitated by each.

(*b*) What are the metals of Group V.?

63. Write out the tests for silver.

64. Write out the tests for mercurous salts.

65. (*a*) Write out the tests for lead.

(*b*) How is lead chloride distinguished from the chlorides of silver and mercury?

(*c*) Give two methods for separating lead from silver and mercury.

66. How can you separate the metals in a solution containing silver, mercury, and lead?

67. (*a*) What happens when H_2S is added to a solution of a mercuric salt?

(*b*) What is "white precipitate"?

68. Why does lead occur both in the silver and copper groups?

69. (*a*) What is the action of water on salts of bismuth?

(*b*) How is lead chromate distinguished from bismuth chromate?

70. (*a*) What is the action of KCN and of H_2SO_4 upon copper sulphide?

(*b*) What happens when $(NH_4)HO$ is added to a solution of copper sulphate?

71. (*a*) What is the action of KCN and of H_2SO_4 upon cadmium sulphide?

(*b*) Give two methods for the separation of copper from cadmium.

72. (*a*) Why is the filtrate from the silver group boiled down with hydrochloric acid before precipitating with H_2S?

(*b*) What is the action of $(NH_4)_2S_2$ on the sulphides of the following metals — lead, antimony, copper, bismuth, tin, and cadmium?

(*c*) What is the action of nitric acid upon sulphides of copper, mercury, bismuth, and cadmium?

73. (*a*) What reactions take place when mercuric chloride is added to stannic chloride and stannous chloride?

(*b*) How is purple of Cassius obtained?

74. (*a*) What is the reaction of zinc upon an acid solution of antimony contained in a platinum vessel?

(*b*) Describe Marsh's test for antimony.

(*c*) What is the action of sulphuretted hydrogen upon an antimony mirror?

75. (*a*) What is the action of silver nitrate upon an acid, neutral, and alkaline solution of arsenious acid?

(*b*) Give the formula of Scheele's green, and state how it may be obtained.

(*c*) Describe Marsh's test for arsenic, and point out how it serves to distinguish that metal from antimony.

(*d*) Describe Reinsch's test for arsenic.

(*e*) Mention several ways of distinguishing an antimony from an arsenic mirror.

(*f*) State all the dry reactions for arsenic.

76. (*a*) What is the action of ammonium carbonate upon the sulphides of arsenic, antimony, and tin?

(*b*) How is tin separated from antimony?

77. (*a*) How is Prussian blue obtained, and what is its formula?

(*b*) How is Turnbull's blue obtained?

(*c*) How are ferrous and ferric salts distinguished by the ferro- and ferri-cyanides of potassium?

78. (*a*) When a solution of the metals of the iron group is precipitated with ammonium sulphide, what causes the filtrate to have a brown colour?

(*b*) What are the blow-pipe tests for nickel?

79. (*a*) What is the action of potassium cyanide upon solutions of nickel and cobalt?

(*b*) How would you separate a solution of the chlorides of nickel and cobalt?

(*c*) What is the action of potassium nitrite upon solutions of nickel and cobalt?

80. How can a solution of zinc hydrate in caustic potash be re-precipitated?

81. (*a*) How can a solution of aluminium hydrate in caustic potash be re-precipitated?

(*b*) How can you distinguish between the hydrate and phosphate of aluminium?

82. (*a*) What happens when ammonia is added to a solution of manganese containing ammonium chloride?

(*b*) What substance is formed when manganese compounds are fused with a mixture of potassium nitrate and carbonate?

(*c*) How would you separate a solution containing the nitrates of iron, zinc, and manganese?

83. (*a*) How would you separate analytically a solution of nickel, cobalt, and chromium?

(*b*) How would you separate analytically a solution containing iron, zinc, chromium, and manganese?

84. (*a*) What is the action of hydrochloric acid upon the sulphides of iron, zinc, nickel, manganese, and cobalt?

(*b*) What is the action of barium carbonate upon a cold solution of each of the following metals—iron, nickel, zinc, chromium, and manganese?

(*c*) How is aluminium distinguished from zinc?

85. Mention three characteristic tests for barium.

86. How is strontium distinguished from barium?

87. How are strontium and calcium distinguished from each other?

88. (*a*) What is the action of strong alcohol upon the chlorides of barium and strontium, and upon the nitrates of strontium and calcium?

(*b*) How are barium, strontium, and calcium separated from each other?

(*c*) What is the action of acetic acid upon the chromates of barium and strontium?

89. What are the tests for magnesium?

90. What is precipitated on adding platinum chloride to potassium chloride? Give its formula.

91. (*a*) What is the action of platinum chloride upon ammonium chloride? Give its formula.

(*b*) How are ammonium salts distinguished from those of potassium?

92. How are potassium salts distinguished from those of sodium?

93. Describe how magnesium, potassium, sodium, and ammonium are separated.

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95. Write out the acids which occur in each of the four groups into which inorganic acids are divided.

96. How are sulphates detected by the blow-pipe?

97. What happens when sulphuric acid is heated with silico-fluorides?

98. (*a*) How does sodium phosphate behave with solutions of the following reagents—magnesium sulphate, lead acetate, ferric chloride, and ammonium molybdate?

(*b*) How is phosphoric acid separated from solutions of phosphates insoluble in water?

(*c*) Give the distinguishing tests for ortho-, pyro-, and meta-phosphoric acids.

99. How is borax recognized by a flame reaction?

100. (*a*) What reaction takes place when calcium chloride solution is added to an oxalate, and what happens when the resulting substance is ignited?

(*b*) How are oxalates distinguished from phosphates?

101. What is the most characteristic test for fluorides?

102. What is the most characteristic test for carbonates?

103. How is silicic acid obtained from a soluble silicate, and how is it converted into silicon dioxide?

104. State how chromic, arsenious, sulphurous, iodic, and arsenic acids are detected in the examination for bases.

105. How is sulphurous converted into hydrosulphuric acid?

106. How are sulphites distinguished from hyposulphites?

108. How is arsenious distinguished form arsenic acid?

109. What is the effect of heat upon iodates, and upon iodic acid?

110. What happens when sulphur dioxide is passed through a solution of potassium chromate?

111. How is chromium oxychloride prepared from common salt and potassium bichromate?

112. (*a*) How is bromine liberated from its compounds, and how is its presence recognized?

(*b*) What happens when bromides are distilled with manganese dioxide and sulphuric acid?

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(*b*) How can you detect chlorides, bromides, and iodides in presence of each other?

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(*b*) How may ammonium sulpho-cyanide be obtained from sodium cyanide, and from mercuric cyanide?

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(*b*) Give equations for the reactions which occur when hydrochloric acid and sulphuric acid act upon sodium hypochlorite.

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117. How is free sulphuretted hydrogen obtained from sulphides, and how is it recognized?

118. Give all the reactions for nitric acid.

119. (*a*) How is chloric distinguished from nitric acid?

(*b*) How is anhydrous perchloric acid obtained?

(*c*) How are perchlorates distinguished from chlorates?

120. (*a*) How is arsenious separated from phosphoric acid?

(*b*) Name the acids whose silver salts are insoluble in nitric acid.

(*c*) How are acids detected in insoluble bodies?

(*d*) How can barium sulphate be decomposed?

121. State the group reagents of the organic acids, and show how they are divided into groups.

122. What is the action of caustic potash upon calcium tartrate?

123. How is tartaric distinguished from citric acid? Give two methods.

125. How are succinic and benzoic acids distinguished?

127. What is the difference between the action of silver nitrate upon ferro- and upon ferri-cyanides?

129. Name the distinguishing tests for formic and acetic acids.

130. How is a soap formed from stearic acid, and how may the fat be separated?

131. What reactions are produced by ferric chloride, and iodic acid upon morphine?

132. How is morphine separated from meconic acid?

133. Name the characteristic tests for strychnine.

134. How is brucine distinguished from strychnine?

136. How are quinine and cinchonine distinguished from each other?

137. Name the tests for uric acid.

138. Name the tests for urea.

140. How is cane sugar distinguished from grape sugar, and how may it be converted into the latter?

141. Name the tests for albumin.

142. What is the most characteristic test for starch?

144. How are tannic and gallic acids distinguished from each other?

145. How are carbon, hydrogen, and nitrogen detected in organic bodies?

146. (*a*) How would you separate a mixture of citric, tartaric, and oxalic acids?

(*b*) How would you separate a mixture of citric, benzoic, and formic acids?

148. Name the rare metals precipitated in the silver, copper, arsenic, iron, and potassium groups.

149. Name the tests for tungsten.

150. Give the formula of the substance produced by adding platinum chloride to solutions of thallium.

151. What reactions are produced by iodide and chloride of potassium in palladium solutions?

152. What reaction takes place when caustic potash is added to rhodium salts?

153. What is the most characteristic test for osmium compounds?

155. Name the most characteristic tests for gold.

156. Name the most characteristic tests for platinum.

157. What is the action of ferrous sulphate upon iridium salts?

158. How does sodium phosphate react upon molybdenum salts?

160. How is selenium distinguished from tellurium?

161. What is the blowpipe test for uranium compounds?

163. How is beryllium distinguished from aluminium?

167. How are cerium, lanthanum, and didymium distinguished?

168. What happens when zinc is added to solutions of titanium salts?

169. (*a*) How is ammonium vanadate obtained?

(*b*) What is the action of oxalic acid, sulphur dioxide, and zinc upon acid solutions of vanadium pentoxide?

170. What reaction is produced by sodium phosphate on lithium salts?

172. How are cæsium and rubidium distinguished from each other and from potassium?

INDEX.

A.

ACETIC acid, reactions of, 122, 123
Acids, bases, and salts, 30
Acids, grouping of the inorganic, 97
Albumen, reactions of, 130
Aluminium, reactions of, 85
Ammonia, preparation of, 16
    properties of, 18
    residue from preparation of, 18
Ammonium, reactions of, 95
Ammonium nitrate, preparation of, 19
Antimony, reactions of, 73
Arsenic, dry reactions for, 78
    reactions of, 76
Arsenic acid, reactions of, 107
Arsenious acid, reactions of, 106

B.

BARIUM, reactions of, 90
Benzoic acid, reactions of, 121
Beryllium, reactions of, 145
Bismuth, reactions of, 67
Blow-pipe reactions, 43
    confirmatory tests, 44
    use of, 41
Boric acid, reactions of, 102
Bromine, preparation of, 34
    properties of, 34
Brucine, reactions of, 127
Bunsen's flame reactions, 46

C.

CADMIUM, reactions of, 69
Cæsium, reactions of, 149
Calcium, hypochlorite, preparation of, 31
Calcium, reactions of, 91
Cane sugar, reactions of, 129
Carbolic acid, 124*a*
Carbon, detection of, in organic bodies, 132
Carbonic acid, reactions of, 104
Carbon dioxide, preparation of, 22
    properties of, 23
Carbon monoxide, preparation of, 24
    properties of, 24
Casein, reactions of, 131
Cerium, reactions of, 146
Chloric acid, reactions of, 113
Chlorine bleaching action of, 28, 31
    detection of, in organic bodies, 132
    preparation of, 26
    properties of, 27
Cholesterine, reactions of, 129
Chromic acid, reactions of, 107
Chromium, reactions of, 87
Cinchonine, reactions of, 128
Citric acid, reactions of, 120
Cobalt, reactions of, 84
Copper, reactions of, 68

D.

DIDYMIUM, reactions of, 147

E.

ELEMENTS, table of the, with their symbols and combining weights, Appendix A, 151

F.

FERRI-CYANIC acid, reactions of, 123
Ferric salts, reactions of, 82
Ferro-cyanic acid, reactions of, 123
Ferrous salts, reactions of, 81
Formic acid, reactions of, 124

G.

GALLIC acid, reactions of, 131
Gold, reactions of, 141
Grape sugar, reactions of, 129

H.

HYDRIODIC acid, reactions of, 110
Hydrobromic acid, reactions of, 109
Hydrochloric acid, preparation of, 28
  properties of, 30
  reactions of, 108
Hydrocyanic acid, reactions of, 111
Hydrofluoric acid, preparation of, 35
  properties of, 35
  reactions of, 103
Hydro-fluosilicic acid, reactions of, 99
Hydrogen, detection of, in organic bodies, 132
  preparation of, 9
  properties of, 11, 12
  residue from preparation of, 11
Hydro-sulphuric acid, reactions of, 113
Hypochlorous acid, preparation of, 31
  reactions of, 112
Hypo-sulphurous acid, reactions of, 106

I.

INDIUM, reactions of, 144
Iodic acid, reactions of, 107
Iodine, preparation of, 32
  properties of, 32
  tests for, 33
Iridium, reactions of, 142
Iron, reactions of, 81

L.

LANTHANUM, reactions of, 146
Lead, reactions of, 64, 67
Lithium, reactions of, 148

M.

MAGNESIUM, reactions of, 94
Malic acid, reactions of, 120
Manganese, reactions of, 86
Marsh's test for antimony, 73
  for arsenic, 77
Meconic acid, reactions of, 125
Mercuric salts, reactions of, 66
Mercurous salts, reactions of, 63
Metallic beads, 48
Metallic films, 48
Metals, grouping of the, 60
Metaphosphoric acid, reactions of, 101
Molybdenum, reactions of, 143
Morphine, reactions of, 125

N.

NICKEL, reactions of, 83
Nitrates, tests for, 16
Nitric acid, preparation of, 14
  reactions of, 15, 113
Nitrogen, preparation of, 12
  properties of, 13
  detection of, in organic bodies, 132
Nitrogen dioxide, preparation of, 20
  properties of, 21
Nitrogen monoxide, preparation of, 19
  properties of, 20
Nitrous acid, reactions of, 112

O.

OPIUM solutions, reactions of, 126
Organic acids, grouping of the, 118
Osmium, reactions of, 140
Oxalic acid, reactions of, 102
Oxidation by blow-pipe, 42
Oxidizing flame, 41
Oxygen, combustions in, 5 to 8
  preparation of, from potassium chlorate and manganese dioxide, 4
  from mercuric oxide, 2
  from potassium chlorate, 3
  presence of, in air, 9
  residue from preparation of, 5

P.

PALLADIUM, reactions of, 139
Perchloric acid, reactions of, 114
Phosphoric acid, reactions of, 100
Phosphorus, blow-pipe test for, 54
  detection of, in organic bodies, 133
Platinum, reactions of, 141
  residues, treatment of, Appendix D, 158

Potassium, reactions of, 94
Pyrophosphoric acid, reactions of, 101

Q.

Quinine, reactions of, 127

R.

Rare metals, grouping of the, 137
Reducing flame, 41
Reduction by blow-pipe, 42
Rhodium, reactions of, 139
Rubidium, reactions of, 149
Ruthenium, reactions of, 140

S.

Salicylic acid, 125
Selenium, reactions of, 143
Silicic acid, reactions of, 104
Silver group, metals of the, 63
Silver, reactions of, 63
Silver residues, treatment of, Appendix C, 155
Single salts, preliminary examination of, 55
Sodium bromide, preparation of, 35
  properties of, 35
Sodium hydrate, preparation of, 39
  properties of, 40
Sodium iodide, preparation of, 33
  properties of, 33
Sodium, reactions of, 95
Stannic salts, reactions of, 72
Stannous salts, reactions of, 72
Starch, reactions of, 131
Stearic acid, reactions of, 124
Stirring rods, 1
Strontium, reactions of, 90
Strychnine, reactions of, 126
Succinic acid, reactions of, 121
Sulpho-cyanic acid, reactions of, 123
Sulphur, blow-pipe test for, 55
Sulphur dioxide, preparation of, 36
Sulphur, detection of, in organic bodies, 133
  properties of, 37
Sulphuretted hydrogen, preparation of, 37
  properties of, 38
Sulphuric acid, reactions of, 99
Sulphurous acid, reactions of, 105

T.

Table A, examination of soluble single salts, 56
  B, examination of insoluble substances, 58
  C, separation of silver group, 65
  D, ,, copper ,, 70
  E, ,, arsenic ,, 80
  F, ,, iron ,, 88
  G, ,, calcium ,, 92
  H, ,, potassium,, 96
  J, detection of inorganic acids, 115
  K, separation of organic acids, 134
  L, iron in presence of phosphate, 156, 157
Table of the elements, with their symbols and combining weights, Appendix A, 151
Tannic acid, reactions of, 131
Tartaric acid, reactions of, 119
Tellurium, reactions of, 143
Thallium, reactions of, 138
Titanium, reactions of, 147
Tungsten, reactions of, 138

U.

Uranium, reactions of, 144
Urea, reactions of, 129
Uric acid, reactions of, 128

V.

Vanadium, reactions of, 148

W.

Wash bottle, 1
Weights and measures, Appendix B, 154

Z.

Zinc, reactions of, 85
Zirconium, reactions of, 145

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